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### CHEMISTRY LABORATORY OPERATIONS MANUAL

SAMUEL SOPOK

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### CHEMISTRY LABORATORY OPERATIONS MANUAL

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#### ABSTRACT

In 1987, using quality as a focal point, an extensive effort was undertaken to research, develop, implement, document, and periodically revise all methods used in SMCAR-CCB-RT's Chemistry Laboratory operations. The resulting information is compiled in this manual and should provide a useful starting point for responsible and efficient operations in the future.

#### KEYWORDS

chemistry laboratory operations.

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#### 1. INTRODUCTION

In 1987, using quality as a focal point, an extensive effort was undertaken to research, develop, implement, document, and periodically revise all methods used in SMCAR-CCB-RT's Chemistry Laboratory operations. The resulting information is compiled in this manual and should provide a useful starting point for responsible and efficient operations in the future.

#### 2. LAB SAFETY

Emergency numbers are: ambulance 5333, chemical spills 5888, and fire 5222.

Hazcom rules require that: Hazcom and hazard control training are understood, Msds's are in the required binders, all missing Msds's are ordered from Industrial Hygiene and updated, received chemicals have Msds's, each chemical storage area has a computerized Hazcom inventory, proper storage areas and container labelling are used, emergency procedures are understood, locations of emergency lab equipment are known, emergency spill procedures are understood, absorbents/neutralizers for spills are maintained, personal protective equipment is used, eye wash and shower stations are inspected weekly, hoods are periodically inspected, and gas cylinders require chains, caps and labels.

Hazardous materials handling rules require that:
hazardous materials handlers have formal training, hazardous
material disposal is done in proper sinks only, separate
storage areas are required for acids (except H2SO4), acids
(only H2SO4), bases, solvents, non-oxidizing solids, oxidizing
solids, buffer solution, and gas cylinders.

General safety rules require that: before use, know how to operate equipment, job safety breakdown sheets are attached to each machine, good housekeeping is used, food is allowed only in office areas, and additional references are in lab book cases.

### 3. DATABASES

### 3.01 HAZARDOUS MATERIALS DATABASE

Database files include: zhazmat.dbf database, zhazmat.\$bf database definition, zhazmat.\$if default input form, and zhazmat.\$rf default report form. The field name, field type,

and field length are: hazmat text 0-30, prodnum text 0-15, manuf text 0-15, manufaddr text 0-30, manuftele phone# 12-12, prodcont text 0-7, gramamt text 0-4, literamt text 0-6, bl15rnum text 0-3, monthusage text 0-6, and msdsdate date 8-8.

A zhazmat default report form example is: hazmat(t):phosphoric acid, prodnum(t):2796-070, manuf(t):mcb, manufaddr(t):paris, ky 40361, manuftele(p):314-982-5000, prodcont(t):glass, gramamt(t): 0, literamt(t): 3.8, Bl15rnum(t):105, monthusage(t): 1, msdsdate(d):85/09/05.

### 3.02 ORDERING DATABASE

Database files include: zorder.dbf database, zorder.\$bf database definition, zorder.\$if default input form, and zorder.\$Rf default report form. The field name, field type, and field length are: item text 0-40, quantity text 0-6, comments text 0-65, formtype text 0-9, provider text 0-20, estcost text 0-10, acct text 0-16, submitted text 0-6, received text 0-6, costarea text 0-6, benetnum text 0-6, contract text 0-9, orderdby text 0-3, and wpfname text 0-12.

A zorder default report form example is: item(t):tru-touch disposable vinyl gloves, quantity(t):1 case, comments(t):large cat no. 55-25002, formtype(t):896, provider(t):krackler scientific, estcost(t):144.50, acct(t):w9hr-4w-001, submitted(t):890426, received(t):890530, costarea(t):hrw, benetnum(t):, contract(t):, orderdby(t):aem, wpfname(t):.

#### 3.03 SAMPLES DATABASE

Database files include: zsamples.dbf database, zsamples.\$bf database definition, zsamples.\$if default input form, zsamples.\$rf default report form, and zsamples.rpt standard report form. The field name, field type, and field length are: zlabnum text 9-9, datecom date 8-8, project number 4-4, charge text 9-9, hours number 3-4, custlnam text 1-12, sampleid text 1-10, samtype text 1-20, analyst text 3-3, datered date 8-8, comment text 0-45, elem1 text 0-20, elem2 text 0-20, elem3 text 0-20, elem4 text 0-20, elem5 text 0-20, elem6 text 0-20, elem7 text 0-20, elem8 text 0-20, elem9 text 0-20, elem10 text 0-20, elem11 text 0-20, elem12 text 0-20, CrO3 number 0-3, H2SO4cp number 0-4, H2SO4sd number 0-4, CRIII number 0-4, Fe number 0-4, H2SO4ep number 0-3, and H3PO4 number 0-3.

A zsamples default report form example is: zlabnum(t):xx0102.01, Datecom(d):xx/01/02, project(n):x.xo, charge(t):xxxxxxxxx, hours(n): 3.0, custlnam(t):xxxxx, sampleid(t):xxxx, samtype(t):4130, analyst(t):xxx,

daterec(d):xx/01/02, comment(t):ASTM 331, elem1(t):C - .308, elem2(t):Mn - .456, elem3(t):P - .015, elem4(t):S - .007, elem5(t):Si - .224, elem6(t):Cu - .200, elem7(t):Ni - .125, elem8(t):Cr - .827, elem9(t):V - .011, elem10(t):Mo - .153, elem11(t):, elem12(t):, CrO3(i):, H2SO4cp(n):, H2SO4sd(n):, CrIII(n):, Fe(n):, H2SO4ep(i):, H3PO4(i):.

A zsamples standard report form example is: chemical analysis report, Advanced Technology Branch, Benet Laboratories, lab number xx0102.01, sampleid xxxx, last name xxxxx, grade/type 4130, date received xx/01/02, date completed xx/01/02, comments ASTM 331, analyte 1 C - .308, analyte 7 Ni - .125, analyte 2 Mn - .456, analyte 8 Cr - .827, analyte 3 P - .015, analyte 9 V - .011, analyte 4 S - .007, analyte 10 Mo - .153, analyte 5 Si - .224, analyte 11, analyte 6 Cu - .200, analyte 12, Cr03 (g/l), CrIII (g/l), Fe (g/l), polishing solutions:, H3PO4 (g/l), H2SO4 (g/l), Cr plating solutions:, H2SO4 (g/l), H2SO4 sd (g/l).

#### 3.04 STANDARD REFERENCE MATERIALS DATABASE

Database files include: zsrm.dbf database, zsrm.\$bf database definition, zsrm.\$if default input form, and zsrm.\$rf default report form.

The field name, field type, and field length are: name text 0-8, type text 0-5, Ag number 0-8, Al number 0-8, As number 0-8, B number 0-8, Be number 0-8, Bi number 0-8, C number 0-8, Ca number 0-8, Cd number 0-8, Ce number 0-8, Co number 0-8, Cr number 0-8, Cu number 0-8, Fe number 0-8, Hf number 0-8, La number 0-8, Mg number 0-8, Mn number 0-8, Mo number 0-8, Nb number 0-8, Nd number 0-8, Ni number 0-8, P number 0-8, P number 0-8, S number 0-8,

A zsamples default report form example is: name(t):1120, type(t):Cu, Ag(n): 0.00000, Al(n): 1.46000, As(n): 0.00000, B(n): 0.00000, Be(n): 0.00000, I(n): 0.00000, C(n): 0.00000, Ca(n): 0.00000, Cd(n): 0.00000, Ce(n): 0.00000, Co(n): 0.00000, Cr(n): 0.00000, Cu(n):80.14000, Fe(n): 0.01500, Hf(n): 0.00000, La(n): 0.00000, Mg(n): 0.00000, Mn(n): 0.00000, Mo(n): 0.00000, Nb(n): 0.00000, Nd(n): 0.00000, Ni(n): 0.00000, Phone of the control of

#### 4. STANDARD ANALYTICAL METHODS

### 4.01 DETERMINATION OF CHROMIC ACID IN CHROMIUM PLATING SOLUTIONS USING A REDOX TITRATION AND INDICATOR

This procedure describes the quantitative analysis of CrO3 in chromium plating solutions using a redox titration and indicator. References: (1, 2).

Glassware includes: 400 ml beakers, 50 ml buret, 10 ml pipet, 25 ml pipet, 500 ml volumetric flasks, 10 ml, and 100 ml graduated cylinders.

Reagent solutions include: 45.0 g/l ferrous ammonium sulfate six hydrate titrant (with 60 ml/l concentrated sulfuric acid), 4.90 g/l potassium dichromate standard, 10.0 g/l sodium diphenylamine sulfonate indicator, concentrated sulfuric acid, concentrated phosphoric acid, and a 250 g/l Cr03 quality control reference sample.

A magnetic stirrer and magnetic stirring bars are needed. Precautions include only normal laboratory practices.

Procedure for standards is three replicates, complete mixing, pipet 25 ml of the chromic acid standard into a 400 ml beaker, and fill the 400 ml beaker to about the 200 ml mark with deionized water.

Procedure for samples is three replicates, complete mixing, pipet 10 ml of a sample solution into a 500 ml volumetric flask and fill to the mark with deionized water, mix flask thoroughly, pipet 25 ml of the diluted sample solution in the flask into a 400 ml beaker, and fill the beaker to about the 200 ml mark with deionized water.

Procedure for standard/sample titration is to add 5 ml of concentrated sulfuric acid to each beaker, add 5 ml of concentrated phosphoric acid to each beaker, add a stirring bar to each beaker, fill buret with ferrous ammonium sulfate solution noting titrant volume, stir standard or sample and add 5 drops of indicator, proceed with the titration until the standard or sample begins to turn purple, then titrate drop by drop until a green endpoint, and record the titrant volume dispensed for each beaker.

The calculation is grams/liter of chromic acid for the sample equals (166.55) (ml of titrant for sample)/(ml of titrant for standard).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remade this reference sample containing only one analyte per solution every three months with a date label. The chromic acid concentration is ideally 240-260 g/l for these solutions.

## 4.02 DETERMINATION OF IRON IN ELECTROPOLISHING AND CHROMIUM PLATING SOLUTIONS BY ATOMIC ABSORPTION SPECTROSCOPY

This procedure describes the quantitative analysis of iron in electropolishing and chrome plating solutions using atomic absorption spectroscopy. References: (1, 3, 4).

Glassware includes: 100 ml volumetric flasks and 0-1 ml micropipet. Reagent solutions include: 1.000 g/l iron standard stock solution (with 50 ml/l of concentrated nitric acid), concentrated nitric acid, and 1.000 g/l iron quality control reference solution (with 50 ml/l concentrated nitric acid).

Atomic absorption spectrometer settings are: 248.3 nm wavelength, 0.2 nm slit size, oxidizing air acetylene flame, 30 mA lamp setting, and 2.00 second integration per reading.

Precautions include making sure that the atomic absorption burner head is filled with deionized water before igniting flame preventing possible flashback.

Procedure for standards is three replicates, complete mixing, micropipet 0.250 ml of iron standard into a 100 ml volumetric flask with 1 ml of concentrated nitric acid, fill this flask to the mark with deionized water, mix this flask thoroughly resulting in a 2.50 ppm iron standard solution, micropipet 0.500 ml of iron standard into a 100 ml volumetric flask with 1 ml of concentrated nitric acid, fill and mix this flask thoroughly as before resulting in a 5.00 ppm iron standard solution, and use deionized water as the standard blank (0.00 ppm iron blank solution).

Procedure for Samples is three replicates, complete mixing, micropipet 0.050 ml of a sample solution into a 100 ml volumetric flask with 1 ml of concentrated nitric acid, 2000 dilution of a sample, fill this flask to the mark with deionized water, and mix thoroughly.

Procedure for atomic absorption readings is record the absorbance readings for each standard and sample using the provided operator's manual reference and assure that the 0.00, 2.50 and 5.00 ppm iron standards are proportional.

The original sample calculation is grams/liter of iron in the sample equals (10) (sample absorbance/5.00 ppm standard absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The iron concentrations are ideally 0-7.5 and 0-15.0 g/l for these chromium plating and electropolishing solutions, respectively.

## 4.03 DETERMINATION OF PHOSPHORIC AND SULFURIC ACIDS IN ELECTROPOLISH SOLUTIONS BY ACID/BASE TITRATION USING A PH METER

This procedure describes the quantitative analysis of phosphoric and sulfuric acids in polishing solutions by acid/base titration using a pH meter. References: (1, 5, 6).

Glassware includes: 10 ml pipets, 250 ml volumetric flasks, 25 ml pipets, 200 ml beakers, and 50 ml buret.

Reagent solutions include: 1.000 normal sodium hydroxide titrant, pH= 4.00 standard buffer one, pH= 10.00 standard buffer two, and a quality control reference sample with 50:50 percent mixture by volume of analytical reagent grade concentrated phosphoric acid and concentrated sulfuric acid each of known individual acid concentrations.

A pH meter, magnetic stirrer, and magnetic stirring bars are needed. Precautions include only normal laboratory practices.

Procedure for standards is not applicable. Procedure for samples is three replicates, cool and mix samples thoroughly, pipet 10 ml of a sample solution into a 250 ml volumetric flask rinsing pipet into flask with deionized water then filling to the mark with deionized water, cool and mix flask thoroughly, pipet 25 ml of the diluted sample solution in the flask into a 200 ml beaker, and fill the beaker to about the 100 ml mark with deionized water.

Procedure for sample titration is add a stirring bar to each beaker, calibrate the pH meter with buffer one (pH=4.00), put pH meter electrodes in the beaker and moderately stir, add the titrant to the buret as needed, dispense titrant from the buret until sample pH is 4.50+-0.10 pH units, record this initial titrant volume in ml dispensed as reading A, calibrate the pH meter with buffer two (pH=10.00), continue titrating until sample pH is 9.70+-0.10 pH units, and record the total titrant volume in ml dispensed as reading B.

The original sample calculations are grams/liter of phosphoric acid in the sample equals (98) (sodium hydroxide titrant normality) (reading B - reading A) and grams/liter of sulfuric acid in the sample equals (49) (sodium hydroxide titrant normality) ( (2 \* reading A) - reading B) ).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample every three months labelling it with the date made. The phosphoric and sulfuric acid concentrations are ideally 640-730 and 795-895 g/l for these respective solutions.

### 4.04 DETERMINATION OF CR(III) ION IN CHROMIUM PLATING SOLUTIONS BY UV-VISIBLE SPECTROSCOPY

This procedure describes the quantitative analysis of Cr(III) ions in chrome plating solutions using uv-visible spectroscopy. References: (1, 7, 8).

Glassware includes: 100 ml volumetric flasks, 5 ml pipet,

25 ml pipet, 50 ml pipet, and matching cells.

Reagent solutions include: 1000 ppm Cr(III) standard stock solution, 250 g/l Cr03 standard stock solution, and 1.000 g/l Cr(III) quality control reference sample as chromium (III) chloride.

The uv-visible spectrometer wavelength is 600 nm. Precautions include only normal laboratory practices.

Procedure for standards is three replicates, mix standard stock solutions completely, prepare standard one by pipetting 5 ml of the chromic acid standard stock solution and 50 ml of the Cr(III) standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix, 0.500 g/l Cr(III) standard solution results, prepare standard two by pipetting 5 ml of the chromic acid standard stock solution and 25 ml of the Cr(III) standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix, 0.250 g/l Cr(III) standard solution results, prepare the standard blank by pipetting 5 ml of the chromic acid standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix, and a 0.000 g/l Cr(III) blank solution results.

Procedure for samples is three replicates, mix samples thoroughly, pipet 5 ml of a sample solution into a 100 ml volumetric flask, and fill this flask to the mark with deionized water and mix giving a 20 dilution of a sample.

Procedure for uv-visible readings is record the absorbance readings for each standard and sample using the provided reference and assure that the 0.000, 0.250 and 0.500 g/l Cr(III) standards are proportional.

The original sample calculation is grams/liter of Cr(III) in the sample equals (10) (sample absorbance/0.500 gram per liter standard absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date made. The Cr(III) concentration is ideally 0-7.5 g/l in these chromium plating solutions.

### 4.05 DETERMINATION OF SULFURIC ACID IN CHROMIUM PLATING SOLUTIONS BY ION CHROMATOGRAPHY

This procedure describes the quantitative analysis of sulfuric acid in chromium plating solutions by ion chromatography. References: (1, 9, 10).

The detailed method includes the following:

Glassware includes: 1000 ml volumetric flasks, 250 ml volumetric flasks, 0-5 ml micropipet, and 0-1 ml micropipet.

Reagent solutions include: 250 g/l Cr03 standard stock solution (only use EM Science reagent grade Cr03), 2.94 g/l sulfuric acid standard stock solution, 0.38 g/l sodium carbonate eluent solution, sulfuric acid regenerate solution (0.75 ml of concentrated sulfuric acid per liter or 1.4 g/l H2SO4), and a quality control reference sample (2.94 g/l sulfuric acid solution, to provide the proper matrix a 250 g/l chromic acid, and again only use EM Science reagent grade Cr03).

Ion chromatographic system and analytical balance are used. Precautions include only normal laboratory practices.

Procedure for standards is three replicates, mix standard stock solutions completely, prepare standard one by micropipetting one ml of the chromic acid standard stock solution and one ml of the sulfuric acid standard stock solution into a 250 ml volumetric flask, and fill this flask to the mark with deionized water and mix.

Procedure for samples is three replicates, mix sample solutions thoroughly, micropipet one ml of a sample solution into a 250 ml volumetric flask, and fill this flask to the mark with deionized water and mix.

Procedure for ion chromatographic analysis is always fill the autosampler racks with new vials, the first four vials should contain standards for calibration, the remaining vials should alternate standards and samples except the last two which contain a standard and deionized water, place autosampler racks/vials in the autosampler and set autosampler in its run mode, set the controller's schedule to one iteration for equilibration program one, set the controller's schedule to an iteration value equal to the number of autosampler vials inserted for analysis program two, fill the eluent reservoir with sodium carbonate eluent solution and purge the air from the analytical pump leaving the pump in the remote mode, put the conductivity detector and the advanced chromatography module into the remote mode, open the main valve on the argon tank, fill the regenerate reservoir with sulfuric acid regenerate solution, program the integrator with at=1024, ph=1, pt=5000, and mn=0, hit the start button on the controller, wait five minutes and make sure the conductivity reading is between 10-30 uS and the pump pressure is between 200-600 psi in the ready mode, and if there are any problems refer to the specific technical report referenced.

Original sample calculations are done according to the referenced technical report above, this uses the enclosed spreadsheet called Z120.SSF. Any further problems are also referred to the specific technical report above.

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The CRO3 concentration is ideally 240-260 g/l and the H2SO4 concentration is ideally 2.40-2.30 g/l in these chromium plating solutions.

Additional required information is included in the following:

The order of 5 ml sample vials in the autosampler cassettes includes: four standards, six standard/sample#2 pairs, six standard/sample#8 pairs, a standard, and a deionized water.

Initial conditions for the autosampler module include: local/remote: local, run/hold: hold then run, status: search, size: 5 ml, type: sample, inj: 1, tray: in/empty, type: loop, mode: prop, bleed: on, and inj/vial: 1.

Initial conditions for the analytical pump module include: local/remote: remote, start/stop: stop, flow (ml/min): 2.3, pressure limit select low alarm (psi): 100, pressure limit select high alarm (psi): 1000, and valve pressure (psi argon): 100.

Initial conditions for the conductivity detector module include: local/remote: remote, cell: on, auto offset: off, temp. Comp.: 0, and Output range (us): 30. The conductivity detector should be periodically calibrated to 147 us using 0.00100 M potassium chloride.

Initial conditions for the advanced chromatography module include: load/inject: inject, valve a: off, valve b: off, local/remote: remote, Dionex separator column: hpic-ag4, Dionex suppressor column: amms, valve pressure (psi argon): 100, regenerate flow rate (ml/min): 5, and regenerate pressure (psi argon): 6.

Conditions for the computer/controller equilibration program 1 follow. At 0.0 min., conditions are: inject, valve b on, auto-offset off, relays off, end run off. At 5.0 min., conditions are: inject, valve b on, auto-offset off, relays off, end run off. At 30.0 min., conditions are: inject, valve b on, auto-offset off, relays off, and end run on. Eluent flow rate is 2.3 ml/min., eluent port #3 is used, temp. select is zero, valve A is continually off, temp. comp. is 1.7, cond. setting full scale is 30 uS, and ac outlets are all continually off.

Conditions for the computer/controller analysis program 2 follow. At 0.0 min., conditions are: load, valve b on, auto-offset off, relays off, end run off. At 0.1 min., conditions are: load, valve b on, auto-offset off, relays #2, and end run off. At 0.2 min., conditions are: load, valve b

on, auto-offset off, relays off, end run off. At 2.2 min., conditions are: load, valve b on, auto-offset on, relays off, and end run off. At 2.3 min., conditions are: inject, valve b on, auto-offset on, relays #1, and end run off. At 10.0 min., conditions are: inject, valve b on, auto-offset on, relays off, and end run on. Eluent flow rate is 2.3 ml/min., eluent port #3 is used, temp. select is zero, valve a is continually off, temp. comp. is 1.7, cond. setting full scale is 30 uS, and ac outlets are all continually off.

Conditions for the computer/controller halt program h follow. At 0.0 min., conditions are: inject, valve b off, auto-offset off, relays off, and end run on. Eluent flow rate is 0.0 ml/min., no eluent port is selected, temp. select is zero, valve ais continually off, temp. comp. is zero, cond. setting full scale is 30 uS, and ac outlets are all off.

Conditions for the computer/controller analysis schedule follow. At step 1, use 1 iteration of program 1. At step 2, use 30 iteration of program 2. At step 3 or greater, use 1 iteration of program h.

Conditions for the computer/integrator analysis program include: ready, date", time", at=1024, ph=1, pt=5000, fi=1. fe=1. mn=0., press 'enter' to skip entry, file name=", time function value, tt=, method number: mn=0, and end of dialog.

Sample spreadsheet report data for sulfuric acid ion chromatographic analysis follows. Although it is not the case below, if a sample's t-test value is not between +-2.78 then that sample is resampled and reanalyzed.

For std1, sam2-1, std2, sam2-2, std3, sam8-1, std4, and sam8-2: respective peak height replicate one data are 162500 150200 158000 146700 158800 145500 154500 150000, respective peak height replicate two data are 163200 146100 161200 144100 158600 144100 157600 144500, respective peak height replicate three data are 157800 144600 159500 144600 161500 144900 161100 144600, respective mean data are: 161167 146967 159567 145133 159633 144833 157733 146367, and respective standard deviation data are 2936.6 2898.9 1601.0 1379.6 1619.7 702.4 3302.0 3147.0.

For sam2-1, sam2-2, sam8-1, and sam8-2: Pooled sd data are 2917.8 1494.4 1248.3 3225.4, 95% confidence data are 0.1206 0.0624 0.0521 0.1362, conc. 1 (n=3) data are 2.7175 2.7297 2.6938 2.8544, conc. 2 (n=3) data are 2.6319 2.6281 2.6712 2.6956, conc. 3 (n=3) data are 2.6941 2.6654 2.6378 2.6389, conc. mean (n=3) data are 2.6812 2.6744 2.6676 2.7296, and conc. sd (n=3) data are 0.0442 0.0514 0.0282 0.1117.

For sam2 and sam8: t-test (bet +-2.78?) data are +0.170 -0.930, conc. mean (n=6,g/1) data are 2.68 2.70, and conc. sd (n=6,g/1) data are 0.04 0.07.

The spreadsheet cell/formulas pairs include: a16: @avg(a10..A12), a18: @std(a10..A12)\*@sqrt(1.5), b16: @avg(b10..b12), b18: @std(b10..b12)\*@sqrt(1.5), b20: @sqrt(((a18\*\*2)+(b18\*\*2))/2), b22: 2.266\*b20\*2.94/a16, b26: 2.94/a10\*b10, b27: 2.94/a11\*b11, b28: 2.94/a12\*b12, b30:

@avg(b26..b28), b32: @std(b26..b28) \*@sqrt(1.5), b36: 1.225\*(b30-d30)/@sqrt(((b32\*\*2)+(d32\*\*2))/2), b37: @avg(b26..b28,d26..d28), b38: @std(b26..b28,d26..d28), b40: 1.225\*(f30-h30)/@sqrt(((f32\*\*2)+(h32\*\*2))/2), b41: @avg(f26..f28,h26..h28), b42: @std(f26..f28,h26..h28), c16: @avg(c10..c12), c18: @std(c10..c12)\*@sqrt(1.5), d16: @avg(d10..d12), d18: @std(d10..d12) \*@sqrt(1.5), d20: @sqrt(((c18\*\*2)+(d18\*\*2))/2), d22: 2.266\*d20\*2.94/c16, d26: 2.94/c10\*d10, d27: 2.94/c11\*d11, d28: 2.94/c12\*d12, d30: @avg(d26..d28), d32: @std(d26..d28) \*@sqrt(1.5), e16: @avg(e10..e12), e18: @std(e10..e12) \*@sqrt(1.5), f16: @avg(f10..f12), f18: @std(f10..f12) \*@sqrt(1.5), f20: @sgrt(((e18\*\*2)+(f18\*\*2))/2), f22: 2.266\*f20\*2.94/e16, f26: 2.94/e10\*f10, f27: 2.94/e11\*f11, f28: 2.94/e12\*f12, f30: @avg(f26..f28), f32: @std(f26..f28) \*@sqrt(1.5), g16: @avg(g10..g12), g18: @std(g10..g12) \*@sqrt(1.5), h16: @avg(h10..h12), h18: @std(h10..h12) \*@sqrt(1.5), h20: @sqrt(((g18\*\*2)+(h18\*\*2))/2), h22: 2.266\*h20\*2.94/g16, h26: 2.94/g10\*h10, h27: 2.94/g11\*h11, h28: 2.94/g12\*h12, h30: @avg(h26..h28), and h32: @std(h26..h28) \*@sqrt(1.5).

### 4.06 DETERMINATION OF SULFURIC ACID IN ANODIZE AND HARDCOAT SOLUTIONS BY ACID/BASE TITRATION USING A PH METER

This procedure describes the quantitative analysis of sulfuric acid in anodize and hardcoat solutions by acid/base titration using a pH meter. References: (1, 5, 11).

Glassware includes: 10 ml pipets, 400 ml beakers, and a 50 ml buret. Reagent solutions include: 1.000 normal sodium hydroxide titrant, pH= 7.00 standard buffer one, and a quality control reference sample of 98 g/l sulfuric acid solution.

A pH meter, magnetic stirrer, and magnetic stirring bars are needed. Precautions include only normal laboratory practices.

Procedure for standards not applicable. Procedure for samples is three replicates, cool and mix samples thoroughly, pipet 10 ml of a sample solution into a 400 ml beaker and fill the beaker to about the 200 ml mark with deionized water.

Procedure for sample titration is add a stirring bar to each beaker, calibrate the pH meter with buffer one (pH=7.00), put pH meter electrodes in the beaker and moderately stir, add the titrant to the buret as needed, dispense titrant from the buret until sample pH is 7.00+-0.10 pH units, and record this value as reading A which is the dispensed titrant volume in ml.

The original sample calculation is grams/liter of sulfuric acid in the sample equals (9.8) (titrant normality) (reading A).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The sulfuric acid concentrations are ideally 90-150 g/l and 120-150 g/l for these respective anodize and hardcoat solutions.

### 4.07 DETERMINATION OF SODIUM DICHROMATE IN SODIUM DICHROMATE SEALING SOLUTION BY UV-VISIBLE SPECTROSCOPY

This procedure describes the quantitative analysis of sodium dichromate in sodium dichromate sealing solutions using uv-visible spectroscopy. References: (1, 7, 12).

Glassware includes 100 ml volumetric flasks and matching cells. Reagent solutions include 50.0 g/l sodium dichromate standard stock solution and a quality control reference sample of 50.0 g/l sodium dichromate solution.

A uv-visible spectrometer (350 nm wavelength) and a 0 - 0.250 ul micropipet are also required. Precautions include only normal laboratory practices.

Procedure for standards is three replicates, mix standard stock solution completely, prepare standard one by pipetting 100 ul of the sodium dichromate standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix with 50.0 ppm sodium dichromate standard solution resulting, prepare standard two by pipetting 50 ul of the sodium dichromate standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix with a 25.0 ppm sodium dichromate standard solution resulting, and prepare standard three as a blank with a 0.0 ppm sodium dichromate blank standard solution resulting.

Procedure for samples is three replicates, mix samples thoroughly, pipet 100 ul of a sample solution into a 100 ml volumetric flask, and fill this flask to the mark with deionized water and mix giving a 1000 dilution of a sample solution.

Procedure for uv-visible readings is record the absorbance readings for each standard and sample using the provided reference and assure that the 0.0, 25.0 and 50.0 ppm sodium dichromate standards are proportional.

The original sample calculation is grams/liter of sodium dichromate in the sample equals (50) (sample absorbance/standard one absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months

labelling it with the date made. The sodium dichromate concentration is ideally 40.0 - 50.0 g/l in these sealing solutions.

### 4.08 DETERMINATION OF BLACK SANDOZ ALUMINUM COLORING DYE BY UV-VISIBLE SPECTROSCOPY

This procedure describes the quantitative analysis of black Sandoz aluminum coloring dye using uv-visible spectroscopy. References: (1, 7, 12).

Glassware includes 100 ml volumetric flasks and matching cells. Reagent solutions include 12.5 g/l Sandoz Black Dye standard stock solution and a quality control reference sample of 12.5 g/l black dye solution.

A uv-visible spectrometer (600 nm wavelength) and a 0 - 0.250 ul micropipet are also needed. Precautions include only

normal laboratory practices.

Procedure for standards is three replicates, mix standard stock solution completely, prepare standard one by pipetting 200 ul of the black dye standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix with a 25.0 ppm black dye standard solution resulting, prepare standard two by pipetting 100 ul of the black dye standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix with a 12.5 ppm black dye standard solution resulting, and prepare standard three as a blank with a 0.0 ppm black dye blank standard solution resulting.

Procedure for samples is three replicates, mix samples thoroughly, pipet 200 ul of a sample solution into a 100 ml volumetric flask, and fill this flask to the mark with deionized water and mix giving a 500 dilution of a sample solution.

Procedure for uv-visible readings is record the uv-visible absorbance readings for each standard and sample using the provided reference and assure that the 0.0, 12.5 and 25.0 ppm black dye standards are proportional.

The original sample calculation is grams/liter of black dye in the sample equals (12.5) (sample absorbance/standard one

absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date made. The black dye concentration is ideally 10.0 - 12.5 g/l in these dye solutions.

### 4.09 DETERMINATION OF OLIVE SANDOZ ALUMINUM COLORING DYE BY UV-VISIBLE SPECTROSCOPY

This procedure describes the quantitative analysis of olive Sandoz aluminum coloring dye using UV-visible spectroscopy. References: (1, 7, 12).

Glassware includes 100 ml volumetric flasks and matching cells. Reagent solutions include 4.50 g/l Sandoz Olive Dye standard stock solution and a quality control reference sample of 4.50 g/l olive dye solution.

A uv-visible spectrometer (375 nm wavelength) and a 0 - 0.250 ul micropipet are required. Precautions include only normal laboratory practices.

Procedure for standards is three replicates, mix standard stock solution completely, prepare standard one by pipetting 200 ul of the olive dye standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix with a 9.00 ppm olive dye standard solution resulting, prepare standard two by pipetting 100 ul of the olive dye standard stock solution into a 100 ml volumetric flask, fill this flask to the mark with deionized water and mix with a 4.50 ppm olive dye standard solution resulting, and prepare standard three as a blank with a 0.0 ppm olive dye blank standard solution resulting.

Procedure for samples is three replicates, mix samples thoroughly, pipet 200 ul of a sample solution into a 100 ml volumetric flask, and fill this flask to the mark with deionized water and mix giving a 500 dilution of a sample solution.

Procedure for uv-visible readings is record the absorbance readings for each standard and sample using the provided reference and assure that the 0.0, 4.50 and 9.00 ppm olive dye standards are proportional.

The original sample calculation is grams/liter of olive dye in the sample equals (4.50) (sample absorbance/standard one absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The olive dye concentration is ideally 3.00 - 4.50 g/l in these dye solutions.

### 4.10 DETERMINATION OF COPPER IN COPPER CYANIDE PLATING SOLUTIONS BY ATOMIC ABSORPTION SPECTROSCOPY

This procedure describes the quantitative analysis of copper in copper cyanide plating solutions using atomic absorption spectroscopy. References: (1, 3, 13).

Glassware includes 500 ml volumetric flasks. Reagent solutions include 1.000 g/l copper standard stock solution and a quality control reference sample of 1.000 g/l copper solution.

An atomic absorption spectrometer (324.8 nm wavelength, 0.7 nm slit size, oxidizing air acetylene flame, 25 mA lamp setting, 2.00 second integration per reading), 0 - 5 ml micropipet, and a 0 - 0.250 ml micropipet are required. Precautions include making sure that the atomic absorption burner head is filled with deionized water before igniting flame preventing possible flashback. Also, Do Not Acidify Cyanides.

Procedure for standards is three replicates, mix standard stock solution completely, make copper standard one by micropipetting 2.50 ml of copper standard into a 500 ml volumetric flask filling the flask to the mark with deionized water giving 5.00 ppm copper, make copper standard two by micropipetting 1.25 ml of copper standard into a 500 ml volumetric flask filling the flask to the mark with deionized water giving 2.50 ppm copper, and make copper standard three blank by using deionized water giving 0.00 ppm copper.

Procedure for samples is three replicates, mix samples thoroughly, micropipet 0.050 ml of a sample solution into a 500 ml volumetric flask, and fill to the mark with deionized water and mix giving a 10000 dilution of a sample.

Procedure for atomic absorption readings is record the absorbance readings for each standard and sample using the provided atomic absorption operators manual reference and assure that the 0.00, 2.50 and 5.00 ppm copper standards are proportional.

The original sample calculation is grams/liter of copper in the original solution equals (50) (sample absorbance/standard one absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The copper concentration is ideally 16.6 - 35.1 g/l in these copper cyanide plating solutions.

### 4.11 DETERMINATION OF CADMIUM IN CADMIUM CYANIDE PLATING SOLUTIONS BY ATOMIC ABSORPTION SPECTROSCOPY

This procedure describes the quantitative analysis of cadmium in cadmium cyanide plating solutions using atomic absorption spectroscopy. References: (1, 3, 13).

Glassware includes 500 ml volumetric flasks. Reagent solutions include 1.000 g/l cadmium standard stock solution and a quality control reference sample of 1.000 g/l cadmium solution.

An atomic absorption spectrometer (228.8 nm wavelength, 0.7 nm slit size, oxidizing air acetylene flame, 8 mA lamp setting, 2.00 second integration per reading), 0-1 ml micropipet, and a 0-0.250 ml micropipet are required.

Precautions include making sure that the atomic absorption burner head is filled with deionized water before igniting flame preventing possible flashback. Also, Do Not Acidify Cyanides.

Procedure for standard is three replicates, mix standard stock solution completely, micropipet 1.00 ml of cadmium standard into a 500 ml volumetric flask filling the flask to the mark with deionized water with resultant solution as 2.00 ppm cadmium standard one, micropipet 0.500 ml of cadmium standard into a 500 ml volumetric flask filling the flask to the mark with deionized water with resultant solution as 1.00 ppm cadmium standard two, and use deionized water blank with resultant solution as 0.00 ppm cadmium standard three blank.

Procedure for samples is three replicates, mix samples thoroughly, micropipet 0.050 ml of a sample solution into a 500 ml volumetric flask, and fill to the mark with deionized water and mix giving a 10000 dilution of a sample.

Procedure for atomic absorption readings is record the absorbance readings for each standard and sample using the provided atomic absorption operators manual reference and assure that the 0.00, 1.00 and 2.00 ppm cadmium standards are proportional.

The original sample calculation is grams/liter of cadmium in the original solution equals (20) (sample absorbance/standard one absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The cadmium concentration is ideally 17.6 - 21.7 g/l in these cadmium cyanide plating solutions.

# 4.12 DETERMINATION OF SODIUM CYANIDE IN COPPER AND CADMIUM CYANIDE PLATING SOLUTIONS USING PRECIPITATION—FORMATION TITRATION

This procedure describes the quantitative analysis of sodium cyanide in copper and cadmium cyanide plating solutions using precipitation-formation titration. References: (1, 14).

Glassware includes 400 ml beakers and a 50 ml buret. Reagent solutions include: 50 g/l sodium cyanide standard (with 1.0 g/l sodium hydroxide solution freshly prepared), 17.0 g/l silver nitrate titrant solution freshly prepared, potassium iodide reagent powder indicator, concentrated ammonium hydroxide, and a quality control reference sample of 50.0 g/l sodium cyanide (with 1.0 g/l sodium hydroxide solution freshly prepared).

A 0 - 5 ml micropipet, analytical balance, magnetic stirrer, and magnetic stirring bars are needed. Precautions include Do Not Acidify Cyanides.

Procedure for standards is three replicates, mix standard completely, pipet 2 ml of the sodium cyanide standard solution into a 400 ml beaker, fill the 400 ml beaker to about the 100 ml mark with deionized water, add 0.1 grams of potassium iodide indicator and 5 ml of ammonium hydroxide to the beaker. Procedure for samples is the same procedure as used for the standards.

Procedure for standard/sample titration is add a stirring bar to each beaker, titrate using the silver nitrate titrant to a faint yellow turbid permanent endpoint, and record the titrant volume dispensed for each beaker.

The calculation is grams/liter of sodium cyanide for the original sample solution equals (50) (ml of titrant for sample)/(ml of titrant for standard).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The sodium cyanide concentration is ideally 31.0 - 57.7 g/l for copper cyanide plating solutions and 80.9 - 148.3 g/l for cadmium cyanide plating solutions in these solutions.

### 4.13 DETERMINATION OF NICKEL IN NICKEL SULFAMATE PLATING SOLUTIONS BY ATOMIC ABSORPTION SPECTROSCOPY

This procedure describes the quantitative analysis of nickel in nickel sulfamate plating solutions using atomic absorption spectroscopy. References: (1, 3, 15).

Glassware includes 100 ml volumetric flasks. Reagent solutions include a 1.000 g/l nickel standard stock solution

and a quality control reference sample of 1.000 g/l nickel solution.

An atomic absorption spectrometer (232.0 nm wavelength, 0.2 nm slit size, oxidizing air acetylene flame, 25 mA lamp setting, 2.00 second integration per reading), 0-1 ml micropipet, and a 0-0.250 ml micropipet are also needed. Precautions include making sure that the atomic absorption burner head is filled with deionized water before igniting flame preventing possible flashback.

Procedure for standards is three replicates, mix standard stock solution completely, micropipet 0.500 ml of nickel standard into a 100 ml volumetric flask filling the flask to the mark with deionized water with a resultant solution as 5.00 ppm nickel standard one, micropipet 0.250 ml of nickel standard into a 100 ml volumetric flask filling the flask to the mark with deionized water with a resultant solution as 2.50 ppm nickel standard two, and use deionized water blank with a resultant solution as 0.00 ppm nickel standard three blank.

Procedure for samples is three replicates, mix samples thoroughly, micropipet 0.100 ml of a sample solution into a 100 ml volumetric flask, and fill to the mark with deionized water and mix giving a 1000 dilution of a sample.

Procedure for atomic absorption readings is record the absorbance readings for each standard and sample using the provided atomic absorption operators manual reference and assure that the 0.00, 2.50 and 5.00 ppm nickel standards are proportional.

The original sample calculation is grams/liter of nickel in the original solution equals (5) (sample absorbance/standard one absorbance).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The nickel concentration is ideally 3.75-4.75~g/l in these nickel sulfamate plating solutions.

### 4.14 DETERMINATION OF TOTAL CYANIDE IN WASTE SOLUTIONS USING PRECIPITATION FORMATION TITRATION

This procedure describes the quantitative analysis of total cyanide in waste solutions using precipitation-formation titration. References: (1, 16).

Glassware includes: cyanide analysis glassware kit, 250 ml volumetric flask, 25 ml pipets, 250 ml erlenmeyer flasks, 0-5 ml micropipet, 50 ml buret, and round bottom flasks.

Reagent solutions include: 50 g/l sodium hydroxide solution, 50% by volume sulfuric acid solution, 50 g/l

magnesium chloride solution, 0.250 g/l silver nitrate standard titrant solution (prepared freshly for each use), concentrated ammonium hydroxide, potassium iodide powder, and a quality control reference sample of 5.00 ppm total cyanide solution prepared freshly for each use from sodium cyanide.

Number 40 weight filter paper, analytical balance, magnetic stirrer, magnetic stirring bars, cyanide analysis apparatus, chlorine test papers, ascorbic acid powder, spatula, and a pH meter are also needed.

Precautions include Do Not Acidify Cyanide Sample Solutions and refrigerate cyanide waste samples before

analysis.

Procedure for sample distillation is clean and tightly assemble all glassware, test cyanide waste solution using the above chlorine test paper, neutralize all chlorine with ascorbic acid powder as indicated by retesting with the test paper, add an additional half spatula of ascorbic acid powder and mix the solution well, filter (#40 paper) the sample into a 250 ml graduated cylinder, fill the "gas scrubber" with the above 50 g/l sodium hydroxide solution to the 50 ml mark and adjust the hot water vacuum so that the fritted disc bubbles moderately, adjust cold water for the condenser to a moderate flow rate, add 250 ml of the filtered sample to the roundbottomed flask and turn on the stirrer, add 50 ml of the above 50% (v/v) sulfuric acid solution through the thistle tube making sure the vacuum is present, add 20 ml of the above 50 q/l magnesium chloride solution to the thistle tube rinsing the tube with a wash bottle, turn the heat setting on "7", boil and reflux the solution in the round bottom flask for one hour, turn the heat off and cool the system for fifteen minutes, dump and rinse the solution in the "gas scrubber" into a 250 ml volumetric flask and fill to the mark with deionized water, and clean all the glassware associated with the distillation system.

Procedure for sample titration is three replicates, pipet 25 ml of the diluted "gas scrubber" sample into a 250 ml erlenmeyer flask adding 4 ml of the above concentrated ammonium hydroxide solution to this flask, also add to the flask 0.1 grams of the above potassium iodide power stirring the flask to dissolve this power, dilute the solution in the flask to about the 100 ml mark, and using no magnetic stirrer titrate the sample solution in the erlenmeyer flask to the first sign of opalescence (precipitate).

The original sample calculation is ppm total cyanide for the original sample solution equals (ml of titrant used) (titrant conc. in g/1) (19.30).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The total cyanide concentration is ideally below five parts per million in these waste solutions.

### 4.15 DETERMINATION OF SOLID METAL SAMPLES BY DIRECT READING EMISSION SPECTROSCOPY

This procedure describes the quantitative analysis of solid metal samples by direct reading emission spectrometry. References: (1, 17, 18, 19).

Glassware is not required. Reagent standard reference materials include a wide array of standard solid reference metal materials and also a wide array of quality control solid metal reference samples.

An Angstrom direct reading emission spectrometer and a belt sander are needed. Precautions include careful use of the belt sander and safety glasses.

Procedure for standards is three replicates, prepare the surface of the standards for a given matrix using the belt sander and standardize the matrix by running each standard.

Procedure for samples is three replicates, prepare the surface of the samples for a given matrix using the belt sander and analyze the samples for this matrix by running each sample including the reference sample.

Procedure for instrument readings is all sample results are calculated by the instrument and a printout for each sample results.

The original sample calculations are all done by the instrument and a printout for each sample results.

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample. The metal sample results are compared to the Society of Automotive Engineers publication above for material acceptance.

### 4.15.01 CHANNEL CONFIGURATION AND SETTINGS

Angstrom V70 vacuum emission spectrometer channel configuration and settings include: "element, wavelength (vacuum in A), channel, attenuator", "P, 1782.87, 19, 08", "S, 1807.31, 03, 07", "B, 1826.40, 11, 11", "Sn, 1899.89, 23, 04", "C, 1930.90, 01, 05", "Se, 1960.90, 15, 07", "As, 1972.62, 09, 04", "Mo, 2020.96, 35, 07", "W, 2079.78, 17, 04", "Cu, 2136.66, 29, 04", "Cu, 2196.46, 39, 07", "Ni, 2254.56, 07, 02", "Co, 2286.87, 13, 02", "Sb, 2312.18, 25, 02", "As, 2350.56, 31, 10", "Be, 2495.49, 55, 04", "Si, 2516.87, 05, 02", "Al, 2568.76, 27, 06", "Al, 2661.18, 54, 03", "Cr, 2677.95, 21, 03", "Fe, 2740.36, 43, 03", "Fe, 2768.32, 37, 03", "Mg, 2791.61, 45, 01", "Cr, 2871.27, 49, 05", "Mn, 2933.91, 41, 04", "Cu, 2962.03, 47, 15", "Sn, 3035.00, 48, 04", "Zn, 3072.92, 33, 07", "Cu, 3109.50, 57, 05", "Nb, 3195.90, 56, 05", "Pb, 3221.47, 42, 12", "Ti, 3242.92, 38, 05", "V, 3280.79, 46, 08", "Ta, 3312.11, 34, 06", "Zn,

3346.53, 52, 01", "Ag, 3383.86, 30, 05", "Zr, 3439.21, 51, 08", "Ni, 3516.05, 40, 02", "Cd, 3611.53, 36, 06", "Hg, 3651.18, 26, 10", "Pb, 3684.52, 24, 03", "Mo, 3865.20, 53, 09", "Si, 3906.63, 22, 09", "Al, 3945.15, 32, 03", "Ca, 3969.59, 06, 07", "W, 4009.88, 44, 08", "Pb, 4058.96, 08, 03", "Sr, 4078.86, 14, 01", "U, 4096.91, 20, 07", "La, 4124.40, 18, 01", "Mo, 4144.71, 12, 09", "Ce, 4187.78, 28, 02", "Ta, 4207.06, 10, 09", "Pr, 4224.16, 02, 06", "Cr, 4255.54, 16, 06", "V, 4380.47, 04, 07".

### 4.15.02 STAINLESS/MARAGING STEEL MATRIX ONE

Matrix Number? \*1 general data includes: Listing=Y, #1:Matrix Number 1, #2:Matrix Class 0, #3:Matrix Label Stainless & Maraging Steels, #4:Ref Chan # 37, #5:Closure #'s 0, #6:F1,Pr, Int Times 50 120 100, #7:Std Smpl Names 1265 86D 88D 90D 93D 1154 1170B 14935G, #8:Dilute Matrix? Y, #9:Print Ref Elmt? Y, #10:Chan Prnt Order 1 41 19 3 5 29 7 49 4 35 32 13 38 23 56 11 15.

Channel #1 C data includes: #15:Pmt Number 1, #16:Hi,Lo Std #'s 5 1, #17:High Std V/R 16696.00, #18:Low Std V/R 1139.000, #19:Print Format 1.3, #20:Slope 1.833813, #21:Intercept -275.3606, #22:Coefficients -0.7357401E-01 0.7753154E-04 -0.2993778E-08 0.1855684E-12 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 41 Mn data includes: #15:Pmt Number 41, #16:Hi,Lo Std #'s 2 1, #17:High Std V/R 11346.00, #18:Low Std V/R 231.0000, #19:Print Format 1.3, #20:Slope 1.379683, #21:Intercept 25.48610, #22:Coefficients -0.9012438E-01 0.1988349E-03 -0.1061631E-07 0.1718905E-11 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 19 P data includes: #15:Pmt Number 19, #16:Hi,Lo Std #'s 6 1, #17:High Std V/R 9813.000, #18:Low Std V/R 1355.000, #19:Print Format 0.3, #20:Slope 1.855831, #21:Intercept 77.23511, #22:Coefficients -0.4824743E-01 0.1600284E-04 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 5 Si data includes: #15:Pmt Number 5, #16:Hi,Lo Std #'s 2 1, #17:High Std V/R 22923.00, #18:Low Std V/R 2052.000, #19:Print Format 1.3, #20:Slope 2.148136, #21:Intercept 89.45264, #22:Coefficients 0.2378747E-02 -0.1380483E-04 0.6203940E-08 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

```
Channel # 29 Cu data includes: #15:Pmt Number 29,
#16:Hi,Lo Std #'s 3 1, #17:High Std V/R 32830.00, #18:Low Std
V/R 1234.000, #19:Print Format 1.3, #20:Slope 2.209999,
#21:Intercept -2.311768, #22:Coefficients -0.2190852E-01
0.1328180E-04 0.3624899E-08 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 7 Ni data includes: #15:Pmt Number 7, #16:Hi, Lo
Std #'s 2 1, #17: High Std V/R 17044.00, #18: Low Std V/R
282.0000, #19:Print Format 2.3, #20:Slope 1.014179,
#21:Intercept -5.031830, #22:Coefficients -0.3074562
0.1410460E-02 -0.6906041E-06 0.2319191E-09 -0.2176608E-13
0.6591849E-18, #23:Number of Iac's 0, #24:Number of Imc's 0.
Channel # 49 Cr data includes: #15:Pmt Number 49,
#16:Hi,Lo Std #'s 2 1, #17:High Std V/R 37518.00, #18:Low Std
V/R 987.0000, #19:Print Format 2.3, #20:Slope 0.9158803, #21:Intercept 35.04230, #22:Coefficients 0.2242231
-0.8549436E-04 0.3246397E-07 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 4 V data includes: #15:Pmt Number 4, #16:Hi,Lo
Std #'s 6 1, #17:High Std V/R 6381.000, #18:Low Std V/R
386.0000, #19:Print Format 1.3, #20:Slope 1.555831,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 35 Mo data includes: #15:Pmt Number 35,
#16:Hi,Lo Std #'s 8 1, #17:High Std V/R 15143.00, #18:Low Std
V/R 357.0000, #19:Print Format 1.3, #20:Slope 0.8312644,
#21:Intercept 18.55075, #22:Coefficients -0.5890022E-01
0.1516308E-03 0.2349715E-07 0.0000000 0.0000000 0.0000000,
#23:Number of Iac's 0, #24:Number of Imc's 0.
     Channel # 32 Al data includes: #15:Pmt Number 32,
#16:Hi,Lo Std #'s 8 1, #17:High Std V/R 18439.00, #18:Low Std
V/R 656.0000, #19:Print Format 0.3, #20:Slope 2.295783,
#21:Intercept 260.8305, #22:Coefficients -0.3222347E-02
0.1720646E-04 0.3550357E-09 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 13 Co data includes: #15:Pmt Number 13,
#16:Hi,Lo Std #'s 8 1, #17:High Std V/R 32153.00, #18:Low Std
V/R 800.0000, #19:Print Format 2.3, #20:Slope 3.083409,
#21:Intercept 412.1498, #22:Coefficients 0.2694258
-0.8064396E-04 0.1303395E-07 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 38 Ti data includes: #15:Pmt Number 38,
#16:Hi,Lo Std #'s 8 1, #17:High Std V/R 22279.00, #18:Low Std
V/R 447.0000, #19:Print Format 1.3, #20:Slope 3.074440,
#21:Intercept 80.63171, #22:Coefficients -0.5712784E-03
0.3893606E-04 0.1366232E-08 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
```

Channel # 23 Sn data includes: #15:Pmt Number 23, #16:Hi, Lo Std #'s 6 1, #17:High Std V/R 13121.00, #18:Low Std V/R 4032.000, #19:Print Format 0.3, #20:Slope 2.187265, #21:Intercept -246.9590, #22:Coefficients 0.7414371E-03 -0.1441024E-05 0.3128191E-09 0.0000000 0.0000000 0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 56 Nb data includes: #15:Pmt Number 56, #16:Hi, Lo Std #'s 6 1, #17:High Std V/R 5782.000, #18:Low Std V/R 935.0000, #19:Print Format 0.3, #20:Slope 1.548264, #21:Intercept 43.24915, #22:Coefficients -0.7651603E-01 0.5241676E-04 0.2868087E-08 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0. Channel # 11 B data includes: #15:Pmt Number 11, #16:Hi,Lo Std #'s 6 1, #17:High Std V/R 9643.000, #18:Low Std V/R 3280.000, #19:Print Format 0.3, #20:Slope 1.854270, #21:Intercept -316.8293, #22:Coefficients -0.6948919E-02 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 15 Se data includes: #15:Pmt Number 15, #16:Hi, Lo Std #'s 7 1, #17:High Std V/R 11115.00, #18:Low Std V/R 1361.000, #19:Print Format 0.3, #20:Slope 0.6014658, #23: Number of Iac's 0, #24: Number of Imc's 0.

#### 4.15.03 ALUMINUM BRONZE COPPER MATRIX TWO

Matrix Number? \*2 general data includes: Listing=Y, # 1:Matrix Number 2, # 2:Matrix Class 0, # 3:Matrix Label Alum-Bronze, # 4:Ref Chan # 39, # 5:Closure #'s 0, # 6:Fl,Pr, Int Times 50 120 100, # 7:Std Smpl Names 1122 1906 1264 Uzhrl, # 8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 23 52 7 37 5 41 54.

Channel # 37 Fe data includes: #15:Pmt Number 37, #16:Hi, Lo Std #'s 2 1, #17:High Std V/R 4902.000, #18:Low Std V/R 489.0000, #19:Print Format 2.3, #20:Slope 1.480046, #21:Intercept -31.48785, #22:Coefficients -0.6780906 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 5 Si data includes: #15:Pmt Number 5, #16:Hi, Lo Std #'s 4 2, #17:High Std V/R 7453.000, #18:Low Std V/R 866.0000, #19:Print Format 1.3, #20:Slope 1.442965, #21:Intercept -4881.359, #22:Coefficients -0.2220914E-02 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 41 Mn data includes: #15:Pmt Number 41, #16:Hi,Lo Std #'s 4 1, #17:High Std V/R 50969.00, #18:Low Std V/R 223.0000, #19:Print Format 1.3, #20:Slope 2.312420, #21:Intercept -1333.374, #22:Coefficients -0.6924496 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 54 Al data includes: #15:Pmt Number 54, #16:Hi,Lo Std #'s 3 1, #17:High Std V/R 4544.000, #18:Low Std V/R 111.0000, #19:Print Format 2.3, #20:Slope 0.7802162, #23: Number of Iac's 0, #24: Number of Imc's 0.

### 4.15.04 GRADE 360/464/332 COPPER MATRIX THREE

Matrix Number? \*3 general data includes: Listing=Y, # 1:Matrix Number 3, # 2:Matrix Class 0, # 3:Matrix Label Copper 360-464-332, # 4:Ref Chan # 39, # 5:Closure #'s 0, # 6:Fl,Pr, Int Times 50 120 100, # 7:Std Smpl Names 1102 1104 9863 9862, # 8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 24 48 33 43 41.

Channel # 48 Sn data includes: #15:Pmt Number 48, #16:Hi,Lo Std #'s 3 1, #17:High Std V/R 1665.000, #18:Low Std V/R 187.0000, #19:Print Format 2.3, #20:Slope 1.725675, #21:Intercept -9.790009, #22:Coefficients -0.2599633 0.1253408E-02 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

#### 4.15.05 GRADE 6000 ALUMINUM MATRIX FOUR

Matrix Number? \*4 general data includes: Listing=Y, # 1:Matrix Number 4, # 2:Matrix Class 0, # 3:Matrix Label Alum 6000 Series, # 4:Ref Chan # 54, # 5:Closure #'s 1,2,5, # 6:F1,Pr, Int Times 50 50 120, # 7:Std Smpl Names 72 408 603 3003, # 8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 5 43 29 41 45 21 52 38. Channel # 5 Si data includes: #15:Pmt Number 5, #16:Hi,Lo Std #'s 3 4, #17: High Std V/R 3440.000, #18: Low Std V/R 1695.000, #19:Print Format 1.3, #20:Slope 1.736695, #21:Intercept 219.3446, #22:Coefficients -0.1349525 #23:Number of Iac's 0, #24:Number of Imc's 0, Channel # 43 Fe data includes: #15:Pmt Number 43, #16:Hi,Lo Std #'s 4 1, #17:High Std V/R 3385.000, #18:Low Std V/R 1291.000, #19:Print Format 1.3, #20:Slope 3.058464, #21:Intercept -10629.38, #22:Coefficients -0.3272527E-01 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 29 Cu data includes: #15:Pmt Number 29, #16:Hi,Lo Std #'s 3 4, #17:High Std V/R 10700.00, #18:Low Std #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 41 Mn data includes: #15:Pmt Number 41, #16:Hi, Lo Std #'s 2 1, #17:High Std V/R 1331.000, #18:Low Std V/R 223.0000, #19:Print Format 1.3, #20:Slope 2.406019, #21:Intercept -716.7472, #22:Coefficients -0.2885640E-01 #23:Number of Iac's 0, #24:Number of Imc's 0. Channel # 45 Mg data includes: #15:Pmt Number 45, #16:Hi, Lo Std #'s 3 2, #17:High Std V/R 4260.000, #18:Low Std V/R 780.0000, #19:Print Format 1.3, #20:Slope 1.095562, #21:Intercept 33.55206, #22:Coefficients -0.2659747E-01 #23: Number of Iac's 0, #24: Number of Imc's 0.

Channel # 21 Cr data includes: #15:Pmt Number 21, #16:Hi,Lo Std #'s 3 4, #17:High Std V/R 3860.000, #18:Low Std V/R 452.0000, #19:Print Format 1.3, #20:Slope 1.299353, #21:Intercept -2503.063, #22:Coefficients -0.3069836E-01 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 52 Zn data includes: #15:Pmt Number 52, #16:Hi, Lo Std #'s 2 4, #17:High Std V/R 195.0000, #18:Low Std V/R 154.0000, #19:Print Format 1.3, #20:Slope 1.519582, #21:Intercept 26.16079, #22:Coefficients -0.1794404 0.1702683E-02 0.0000000 0.0000000 0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 38 Ti data includes: #15:Pmt Number 38, #16:Hi, Lo Std #'s 2 3, #17:High Std V/R 3859.000, #18:Low Std V/R 1733.000, #19:Print Format 1.3, #20:Slope 6.150473, #21:Intercept 278.3735, #22:Coefficients -0.4228699E-01 #23:Number of Iac's 0, #24:Number of Imc's 0.

#### 4.15.06 PURE COPPER MATRIX FIVE

Matrix Number? \*5 general data includes: Listing=Y, # 1:Matrix Number 5, # 2:Matrix Class 0, # 3:Matrix Label Pure Copper, # 4:Ref Chan # 39, # 5:Closure #'s 0, # 6:Fl,Pr, Int Times 50 120 100, # 7:Std Smpl Names 1121 71X21 1367 1118 1678, # 8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 52 24 43 48 7 32 55 41 19 22 30 13. Channel # 52 Zn data includes: #15:Pmt Number 52, #16:Hi,Lo Std #'s 2 1, #17:High Std V/R 6742.000, #18:Low Std V/R 124.0000, #19:Print Format 1.3, #20:Slope 1.424830, #21:Intercept 37.86649, #22:Coefficients -0.1244892 0.9647125E-03 0.0000000 0.0000000 0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 24 Pb data includes: #15:Pmt Number 24, #16:Hi,Lo Std #'s 2 1, #17:High Std V/R 5610.000, #18:Low Std V/R 213.0000, #19:Print Format 1.3, #20:Slope 1.174086, #21:Intercept -16.73682, #22:Coefficients -0.4075212 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 43 Fe data includes: #15:Pmt Number 43, #16:Hi,Lo Std #'s 3 1, #17:High Std V/R 14906.00, #18:Low Std V/R 913.0000, #19:Print Format 1.3, #20:Slope 1.684247, #21:Intercept -164.1033, #22:Coefficients -0.6988963E-01 #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 48 Sn data includes: #15:Pmt Number 48, #16:Hi,Lo Std #'s 2 1, #17:High Std V/R 5630.000, #18:Low Std V/R 166.0000, #19:Print Format 1.3, #20:Slope 1.470671, #21:Intercept -11.80945, #22:Coefficients -0.1650922 #23:Number of Iac's 0, #24:Number of Imc's 0.

```
Channel # 7 Ni data includes: #15:Pmt Number 7, #16:Hi, Lo
Std #'s 3 1, #17:High Std V/R 8154.000, #18:Low Std V/R
284.0000, #19:Print Format 1.3, #20:Slope 1.615696,
#21:Intercept -19.29648, #22:Coefficients -0.1423298
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 32 Al data includes: #15:Pmt Number 32,
#16:Hi,Lo Std #'s 4 1, #17:High Std V/R 11304.00, #18:Low Std
V/R 398.0000, #19:Print Format 1.3, #20:Slope 0.8960825,
#21:Intercept -3.996918, #22:Coefficients -0.1130662
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 55 Be data includes: #15:Pmt Number 55,
#16:Hi,Lo Std #'s 1 5, #17:High Std V/R 2350.000, #18:Low Std
V/R 259.0000, #19:Print Format 1.3, #20:Slope 0.2112692,
#21:Intercept -7.032898, #22:Coefficients -0.8028150E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 41 Mn data includes: #15:Pmt Number 41,
#16:Hi, Lo Std #'s 3 1, #17:High Std V/R 14442.00, #18:Low Std
V/R 219.0000, #19:Print Format 1.3, #20:Slope 2.101663,
#21:Intercept -31.75520, #22:Coefficients -0.5049044E-01
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 19 P data includes: #15:Pmt Number 19,
#16:Hi,Lo Std #'s 4 1, #17:High Std V/R 2513.000, #18:Low Std
V/R 240.0000, #19:Print Format 1.3, #20:Slope 0.9970309,
#21:Intercept -31.22540, #22:Coefficients -0.2324919E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 22 Si data includes: #15:Pmt Number 22,
#16:Hi,Lo Std #'s 1 2, #17:High Std V/R 1159.000, #18:Low Std
V/R 598.0000, #19:Print Format 1.3, #20:Slope 2.018385,
#21:Intercept -99.68115, #22:Coefficients -0.1211476
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 30 Ag data includes: #15:Pmt Number 30,
#16:Hi,Lo Std #'s 1 2, #17:High Std V/R 467.0000, #18:Low Std
V/R 375.0000, #19:Print Format 1.3, #20:Slope 1.413334,
#21:Intercept -232.2474, #22:Coefficients -0.1888999E-01
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 13 Co data includes: #15:Pmt Number 13,
#16:Hi,Lo Std #'s 5 1, #17:High Std V/R 21621.00, #18:Low Std
V/R 4966.000, #19:Print Format 1.3, #20:Slope 5.879215,
#21:Intercept -2226.358, #22:Coefficients -0.3877140
#23: Number of Iac's 0, #24: Number of Imc's 0.
```

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Matrix Number? *6 general data include: Listing=Y, #
1:Matrix Number 6, # 2:Matrix Class 0, # 3:Matrix Label Tool
Steel, # 4:Ref Chan # 37, # 5:Closure #'s 0, # 6:Fl,Pr, Int
Times 50 100 100, # 7:Std Smpl Names 837 840 841 37B 487 1265
93D 94B, # 8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 1 41 19 3 5 7 49 4 35 17 13.
    Channel # 1 C data include: #15:Pmt Number 1, #16:Hi,Lo
Std #'s 4 6, #17:High Std V/R 21894.00, #18:Low Std V/R
1139.000, #19:Print Format 1.3, #20:Slope 3.525887,
#21:Intercept 151.7933, #22:Coefficients -0.9981561E-01
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 41 Mn data include: #15:Pmt Number 41,
#16:Hi,Lo Std #'s 7 6, #17:High Std V/R 4169.000, #18:Low Std
V/R 231.0000, #19:Print Format 1.3, #20:Slope 1.318670,
#21:Intercept -17.88518, #22:Coefficients -0.7338348E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 19 P data include: #15:Pmt Number 19, #16:Hi,Lo
Std #'s 7 6, #17:High Std V/R 4576.000, #18:Low Std V/R
1355.000, #19:Print Format 1.3, #20:Slope 3.470548,
#21:Intercept -121.5925, #22:Coefficients -0.1235373E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 3 S data include: #15:Pmt Number 3, #16:Hi,Lo
Std #'s 4 6, #17:High Std V/R 3215.000, #18:Low Std V/R
1003.000, #19:Print Format 1.3, #20:Slope 5.713789,
#21:Intercept 110.4837, #22:Coefficients -0.1776462E-02
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 5 Si data include: #15:Pmt Number 5, #16:Hi,Lo
Std #'s 8 6, #17: High Std V/R 11823.00, #18: Low Std V/R
2052.000, #19:Print Format 1.3, #20:Slope 2.644172,
#21:Intercept -240.2927, #22:Coefficients -0.1432564
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 7 Ni data include: #15:Pmt Number 7, #16:Hi,Lo
Std #'s 7 6, #17: High Std V/R 908.0000, #18: Low Std V/R
282.0000, #19:Print Format 2.3, #20:Slope 1.537125, #21:Intercept -62.20596, #22:Coefficients -0.1772568
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 49 Cr data include: #15:Pmt Number 49,
#16:Hi,Lo Std #'s 4 6, #17:High Std V/R 22787.00, #18:Low Std
V/R 967.0000, #19:Print Format 2.3, #20:Slope 0.9458532,
#21:Intercept -184.3037, #22:Coefficients -3.480877
#23: Number of Iac's 0, #24: Number of Imc's 0.
```

Channel # 4 V data include: #15:Pmt Number 4, #16:Hi,Lo Std #'s 1 6, #17: High Std V/R 27815.00, #18: Low Std V/R 386.0000, #19:Print Format 1.3, #20:Slope 0.8073403, #21:Intercept 45.08856, #22:Coefficients 0.1336174 -0.3333828E-04 0.5857968E-08 0.0000000 0.0000000 0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 35 Mo data include: #15:Pmt Number 35, #16:Hi, Lo Std #'s 5 6, #17:High Std V/R 18913.00, #18:Low Std V/R 357.0000, #19:Print Format 1.3, #20:Slope 1.903882, #21:Intercept -81.51410, #22:Coefficients 0.4647553E-01 0.2729543E-04 0.3342094E-07 0.0000000 0.0000000 0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 17 W data include: #15:Pmt Number 17, #16:Hi,Lo Std #'s 3 6, #17:High Std V/R 36608.00, #18:Low Std V/R 604.0000, #19:Print Format 2.3, #20:Slope 6.439520, #21:Intercept -219.9949, #22:Coefficients 0.7416248E-01 0.2289429E-04 0.1780762E-07 0.0000000 0.0000000 0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0. Channel # 13 Co data include: #15:Pmt Number 13, #16:Hi, Lo Std #'s 2 6, #17:High Std V/R 36805.00, #18:Low Std V/R 406.0000, #19:Print Format 1.3, #20:Slope 5.048316, #21:Intercept -78.48331, #22:Coefficients 0.3415766 -0.1365931E-03 0.1492725E-07 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

#### 4.15.08 LOW ALLOY STEEL MATRIX SEVEN

Matrix Number? \*7 general data include: Listing=Y, # 1:Matrix Number 7, # 2:Matrix Class 0, # 3:Matrix Label Low Alloy, # 4:Ref Chan # 37, # 5:Closure #'s 0, # 6:Fl,Pr, Int Times 50 120 100, # 7:Std Smpl Names 1265 1261 1262 1263 1264 Brit2 Brit1 72A 1156, # 8:Dilute Matrix? N, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 1 41 19 3 5 29 7 21 4 35 17 9 23 32 56 11 51 38 8 6.

Channel # 1 C data include: #15:Pmt Number 1, #16:Hi, Lo Std #'s 2 1, #17:High Std V/R 4362.600, #18:Low Std V/R 814.0000, #19:Print Format 2.3, #20:Slope 0.9532636, #21:Intercept 259.9030, #22:Coefficients -0.5686675E-01 0.6493137E-04 0.8175194E-08 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 41 Mn data include: #15:Pmt Number 41, #16:Hi,Lo Std #'s 4 1, #17:High Std V/R 7650.000, #18:Low Std V/R 200.0000, #19:Print Format 1.3, #20:Slope 1.390903, #21:Intercept 1.267334, #22:Coefficients 0.1025945E-02 0.9538596E-04 0.1290560E-07 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

```
Channel # 19 P data include: #15:Pmt Number 19, #16:Hi,Lo
Std #'s 3 1, #17:High Std V/R 2706.000, #18:Low Std V/R
807.0000, #19:Print Format 0.3, #20:Slope 1.106120,
#21:Intercept 29.52936, #22:Coefficients -0.6091101E-02
0.9521732E-05 0.2749520E-08 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 3 S data include: #15:Pmt Number 3, #16:Hi, Lo
Std #'s 3 1, #17:High Std V/R 4066.000, #18:Low Std V/R
1362.000, #19:Print Format 0.3, #20:Slope 4.803064,
#21:Intercept -5.786011, #22:Coefficients -0.4647382E-02
0.5835105E-05 0.1090281E-08 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 5 Si data include: #15:Pmt Number 5, #16:Hi, Lo
Std #'s 4 1, #17:High Std V/R 10083.00, #18:Low Std V/R
1650.000, #19:Print Format 1.3, #20:Slope 1.881116,
#21:Intercept 101.4926, #22:Coefficients -0.1151126
0.7611663E-04 -0.2226753E-08 0.3127475E-12 0.0000000
0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 29 Cu data include: #15:Pmt Number 29,
#16:Hi,Lo Std #'s 3 1, #17:High Std V/R 15288.00, #18:Low Std
V/R 1572.000, #19:Print Format 1.3, #20:Slope 2.936084,
#21:Intercept -76.07324, #22:Coefficients -0.2606053E-01
0.1833495E-04 0.9603757E-09 0.5914607E-14 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 7 Ni data include: #15:Pmt Number 7, #16:Hi, Lo
Std #'s 6 1, #17:High Std V/R 2595.000, #18:Low Std V/R
275.0000, #19:Print Format 1.3, #20:Slope 0.9374520,
#21:Intercept 4.952118, #22:Coefficients -0.5439530E-01
0.2494009E-03 0.3090157E-06 -0.2334154E-10 0.0000000
0.0000000, #23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 21 Cr data include: #15:Pmt Number 21,
#16:Hi,Lo Std #'s 4 1, #17:High Std V/R 9121.000, #18:Low Std
V/R 412.0000, #19:Print Format 1.3, #20:Slope 0.8988667,
#21:Intercept -3.939301, #22:Coefficients -0.1890439E-01 0.6226296E-04 0.5070181E-08 0.4508444E-12 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
     Channel # 4 V data include: #15:Pmt Number 4, #16:Hi, Lo
Std #'s 4 1, #17:High Std V/R 5999.000, #18:Low Std V/R
300.0000, #19:Print Format 0.3, #20:Slope 1.819121,
#21:Intercept -39.66367, #22:Coefficients -0.7481941E-02
0.2414884E-04 0.1277036E-07 -0.1329206E-11 0.0000000
0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.
     Channel # 35 Mo data include: #15:Pmt Number 35,
#16:Hi,Lo Std #'s 7 1, #17:High Std V/R 5608.000, #18:Low Std
V/R 307.0000, #19:Print Format 1.3, #20:Slope 0.6564190,
#21:Intercept 32.54660, #22:Coefficients -0.2521233E-01
0.9457887E-04 0.3315628E-07 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
```

```
Channel # 17 W data include: #15:Pmt Number 17, #16:Hi,Lo
Std #'s 3 2, #17:High Std V/R 1457.000, #18:Low Std V/R
489.0000, #19:Print Format 0.3, #20:Slope 2.376364,
#21:Intercept -110.3798, #22:Coefficients -0.1003587
0.2528827E-03 -0.3199313E-07 0.0000000 0.0000000 0.0000000,
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 9 As data include: #15:Pmt Number 9, #16:Hi,Lo
Std #'s 3 1, #17:High Std V/R 6969.000, #18:Low Std V/R
2297.000, #19:Print Format 0.3, #20:Slope 2.472281,
#21:Intercept -169.1765, #22:Coefficients -0.4398994E-01
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 23 Sn data include: #15:Pmt Number 23,
#16:Hi,Lo Std #'s 4 1, #17:High Std V/R 18070.00, #18:Low Std
V/R 2665.000, #19:Print Format 0.3, #20:Slope 1.555685,
#21:Intercept -366.0471, #22:Coefficients -0.1032518E-01
0.3453512E-05 0.1468151E-09 0.0000000 0.0000000 0.0000000,
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 32 Al data include: #15:Pmt Number 32,
#16:Hi,Lo Std #'s 8 1, #17:High Std V/R 2932.000, #18:Low Std
V/R 1542.000, #19:Print Format 0.3, #20:Slope 3.068545,
#21:Intercept 1013.472, #22:Coefficients -0.3517115E-01
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 56 Nb data include: #15:Pmt Number 56,
#16:Hi, Lo Std #'s 3 2, #17:High Std V/R 5545.000, #18:Low Std
V/R 1283.000, #19:Print Format 1.3, #20:Slope 1.806331,
#21:Intercept -278.1096, #22:Coefficients -0.3680198E-01
0.4237898E-04 0.2983660E-08 0.0000000 0.0000000 0.0000000,
#23:Number of Iac's 0, #24:Number of Imc's 0.
    Channel # 11 B data include: #15:Pmt Number 11, #16:Hi,Lo
Std #'s 5 1, #17:High Std V/R 9955.000, #18:Low Std V/R
2205.000, #19:Print Format 0.3, #20:Slope 1.044006,
#21:Intercept 129.5747, #22:Coefficients -0.3231185E-02
#23: Number of Iac's 1: A: Int'f'g Chan 3 Coeffs 23.31433,
-24314.29, 0.0000000, #24:Number of Imc's 0.
     Channel # 51 Zr data include: #15:Pmt Number 51,
#16:Hi,Lo Std #'s 3 1, #17:High Std V/R 16821.00, #18:Low Std
V/R 1780.000, #19:Print Format 0.3, #20:Slope 1.395881,
#21:Intercept -327.0339, #22:Coefficients -0.2231237E-01
0.1135420E-04 0.7558509E-10 0.0000000 0.0000000 0.0000000,
#23:Number of Iac's 0, #24:Number of Imc's 0.
     Channel # 38 Ti data include: #15:Pmt Number 38,
#16:Hi,Lo Std #'s 5 2, #17:High Std V/R 6801.000, #18:Low Std
V/R 1131.000, #19:Print Format 1.4, #20:Slope 3.623934,
#23:Number of Iac's 0, #24:Number of Imc's 0.
```

# 4.15.09 GRADE 2000 ALUMINUM MATRIX EIGHT

Matrix Number? \*8 general data include: Listing=Y, #
1:Matrix Number 8, # 2:Matrix Class 0, # 3:Matrix Label 2000
Alum Series, # 4:Ref Chan # 54, # 5:Closure #'s 1,2,5, # 6:Fl,
Pr, Int Times 50 50 120, # 7:Std Smpl Names 17C 601 24K 602, #
8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order
5 43 39 41 45 21 52.

Channel # 43 Fe data include: #15:Pmt Number 43, #16:Hi,Lo Std #'s 2 4, #17:High Std V/R 3718.000, #18:Low Std V/R 2289.000, #19:Print Format 1.3, #20:Slope 1.404131, #21:Intercept 242.4281, #22:Coefficients -0.1027367 0.1741249E-03 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 39 Cu data include: #15:Pmt Number 39, #16:Hi,Lo Std #'s 4 1, #17:High Std V/R 2452.000, #18:Low Std V/R 2362.000, #19:Print Format 1.3, #20:Slope 2.726537, #21:Intercept -2022.690, #22:Coefficients -8.977431 0.5584016E-02 0.0000000 0.0000000 0.0000000,

#23: Number of Iac's 0, #24: Number of Imc's 0.

Channel # 45 Mg data include: #15:Pmt Number 45, #16:Hi,Lo Std #'s 3 1, #17:High Std V/R 9242.000, #18:Low Std V/R 2851.000, #19:Print Format 1.3, #20:Slope 0.9928861, #21:Intercept -173.7605, #22:Coefficients 0.1461858E-01, 0.2035468E-03, 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

#### 4.15.10 GRADE 660 COPPER MATRIX NINE

Matrix Number? \*9 general data include: Listing=Y, #
1:Matrix Number 9, # 2:Matrix Class 0, # 3:Matrix Label Copper
660, # 4:Ref Chan # 39, # 5:Closure #'s 0, # 6:Fl,Pr, Int
Times 50 120 100, # 7:Std Smpl Names 1113 Ue13 71x21, #
8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order
24 48 52.

Channel # 24 Pb data include: #15:Pmt Number 24, #16:Hi,Lo Std #'s 3 2, #17:High Std V/R 5610.000, #18:Low Std V/R 782.0000, #19:Print Format 2.3, #20:Slope 2.249649, #21:Intercept -150.2813, #22:Coefficients -0.4740782 0.1107360E-02 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 48 Sn data include: #15:Pmt Number 48, #16:Hi,Lo Std #'s 3 1, #17:High Std V/R 5630.000, #18:Low Std V/R 157.0000, #19:Print Format 2.3, #20:Slope 2.058445, #21:Intercept -71.61847, #22:Coefficients -0.5113173E-01 0.1156987E-02 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

Channel # 52 Zn data include: #15:Pmt Number 52, #16:Hi,Lo Std #'s 3 2, #17:High Std V/R 6742.000, #18:Low Std V/R 896.0000, #19:Print Format 2.3, #20:Slope 2.298467, #21:Intercept -162.8921, #22:Coefficients -0.1884544 0.9627028E-03 0.0000000 0.0000000 0.0000000, #23:Number of Iac's 0, #24:Number of Imc's 0.

#### 4.15.11 GRADE 7000 ALUMINUM MATRIX TEN

Matrix Number? \*10 general data include: Listing=Y, # 1:Matrix Number 10, # 2:Matrix Class 0, # 3:Matrix Label Alum 7000 Series, # 4:Ref Chan # 54, # 5:Closure #'s 1,2,5, # 6:Fl,Pr, Int Times 50 50 120, # 7:Std Smpl Names 924 75G 7050 7076, # 8:Dilute Matrix? Y, # 9:Print Ref Elmt? Y, #10:Chan Prnt Order 22 43 39 41 45 21 52 38.

```
Channel # 22 Si data include: #15:Pmt Number 22,
#16:Hi,Lo Std #'s 2 3, #17:High Std V/R 3582.000, #18:Low Std
V/R 2890.000, #19:Print Format 1.3, #20:Slope 3.587217,
#21:Intercept -1511.955, #22:Coefficients -0.7232350
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 43 Fe data include: #15:Pmt Number 43,
#16:Hi,Lo Std #'s 1 3, #17:High Std V/R 3241.000, #18:Low Std
V/R 1261.000, #19:Print Format 1.3, #20:Slope 1.728987,
#21:Intercept -5425.660, #22:Coefficients -0.1967136
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 39 Cu data include: #15:Pmt Number 39,
#16:Hi,Lo Std #'s 3 4, #17:High Std V/R 2050.000, #18:Low Std
V/R 1812.000, #19:Print Format 1.3, #20:Slope 0.4399904,
#21:Intercept 428.9133, #22:Coefficients -14.39365 0.8329540E-02 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 41 Mn data include: #15:Pmt Number 41,
#16:Hi,Lo Std #'s 2 3, #17:High Std V/R 1752.000, #18:Low Std
V/R 605.0000, #19:Print Format 1.3, #20:Slope 2.293244,
#21:Intercept -722.9210, #22:Coefficients -0.2210569E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 45 Mg data include: #15:Pmt Number 45,
#16:Hi,Lo Std #'s 2 4, #17:High Std V/R 14579.00, #18:Low Std
V/R 9161.000, #19:Print Format 1.3, #20:Slope 1.147225,
#21:Intercept -641.8857, #22:Coefficients 0.2091336E-01
#23:Number of Iac's 0, #24:Number of Imc's 0.
Channel # 21 Cr data include: #15:Pmt Number 21, #16:Hi,Lo Std #'s 2 3, #17:High Std V/R 4212.000, #18:Low Std
V/R 690.0000, #19:Print Format 1.3, #20:Slope 1.576095,
#21:Intercept -3925.268, #22:Coefficients -0.2714468E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 52 Zn data include: #15:Pmt Number 52,
#16:Hi,Lo Std #'s 3 1, #17:High Std V/R 4589.000, #18:Low Std
V/R 2693.000, #19:Print Format 1.3, #20:Slope 1.748695,
#21:Intercept -169.6748, #22:Coefficients 0.3862929 0.1427373E-02 0.0000000 0.0000000 0.0000000,
#23: Number of Iac's 0, #24: Number of Imc's 0.
    Channel # 38 Ti data include: #15:Pmt Number 38,
#16:Hi,Lo Std #'s 4 3, #17:High Std V/R 2139.000, #18:Low Std
V/R 1618.000, #19:Print Format 1.3, #20:Slope 7.760298,
#21:Intercept -151.5249, #22:Coefficients -0.4266750E-01
#23: Number of Iac's 0, #24: Number of Imc's 0.
```

# 4.16 DETERMINATION OF SOLID METAL SAMPLES BY AA/ICP SPECTROSCOPY

This procedure describes the quantitative analysis of solid metal samples by AA/ICP spectrometry. References: (1, 3, 18).

Glassware includes various volumetric flasks and various pipets. Reagent solutions or reference materials include a wide array of standard reference metal solutions and a wide array of quality control metal reference sample solutions.

An AA/ICP spectrometer, various acids, and various machining devices are needed. Precautions include careful use of various machines and safety glasses.

Procedure for standards is three replicates, prepare the standard metal solutions for a given method and standardize the method by running each standard.

Procedure for samples is three replicates and prepare the sample metal solutions for a given method by dissolving these samples in an appropriate acid and analyze the samples for this method by running each sample including the reference sample.

Procedure for instrument readings is all results are calculated by the instrument and a printout for each sample results.

Original sample calculations are all done by the instrument and a printout for each sample results.

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake these reference sample solutions every three months. The metal sample results are compared to the Society of Automotive Engineers publication above for material acceptance.

# 4.17 DETERMINATION OF SOLID METAL SAMPLES BY CARBON-SULFUR ANALYZER

This procedure describes the quantitative analysis of solid metal samples by a carbon-sulfur analyzer. References: (1, 18, 20).

No glassware is needed. Reagent solutions or reference materials include a wide array of standard solid reference metal materials and also a wide array of quality control solid metal reference samples.

A Leco carbon-sulfur analyzer, various machining devices, and Leco crucibles are needed. Precautions include careful use of various machining devices and safety glasses.

Procedure for standards is three replicates, prepare the standards for a given matrix using the various reference materials available, and standardize the method by running each standard.

Procedure for samples is three replicates, prepare the samples for a given method using the various machining devices available and analyze the samples for this method by running each sample including the reference sample.

Procedure for instrument readings is all sample results are calculated by the instrument and a printout for each sample results.

Original sample calculations are all done by the instrument and a printout for each sample results.

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample. The metal sample results are compared to the Society of Automotive Engineers publication above for material acceptance.

# 4.18 RAW MATERIAL DETERMINATION OF TECHNICAL GRADE CHROMIC ACID SALT USING A REDOX TITRATION AND INDICATOR

This procedure describes the raw material determination of technical grade CRO3 using a redox titration and indicator. References: (1, 2).

Glassware includes: 400 ml beakers, 50 ml buret, 10 ml pipet, 25 ml pipet, 500 ml volumetric flasks, 10 ml and 100 ml graduated cylinders, and 100 ml volumetric flasks.

Reagent solutions include: 45.0 g/l ferrous ammonium sulfate six hydrate titrant (with 60 ml/l of concentrated sulfuric acid), 4.90 g/l potassium dichromate standard, 10.0 g/l sodium diphenylamine sulfonate indicator, concentrated sulfuric acid, concentrated phosphoric acid, and a quality control reference sample of 250 g/l Cr03 solution.

A magnetic stirrer and magnetic stirring bars are needed. Precautions include only normal laboratory practice.

Procedure for standards is three replicates, mix standard completely, pipet 25 ml of the chromic acid standard into a 400 ml beaker, and fill the 400 ml beaker to about the 200 ml mark with deionized water.

Procedure for samples is three replicates, put 5.000 grams of the technical grade chromic acid salt in a 100 ml volumetric flask and fill to the mark, mix samples thoroughly, pipet 10 ml of a sample solution into a 500 ml volumetric flask and fill to the mark with deionized water, mix flask thoroughly, pipet 25 ml of the diluted sample solution in the flask into a 400 ml beaker, and fill the beaker to about the 200 ml mark with deionized water.

Procedure for standard/sample titration is add 5 ml of concentrated sulfuric acid to each beaker, add 5 ml of concentrated phosphoric acid to each beaker, add a stirring bar to each beaker, fill buret with ferrous ammonium sulfate solution noting titrant volume, stir standard or sample and add 5 drops of indicator, proceed with the titration until the

standard or sample begins to turn purple, then titrate drop by drop until a green endpoint, and record the titrant volume dispensed for each beaker.

The calculation is weight percent of chromic acid for the sample equals  $(100/50)\,(166.55)\,(\text{ml of sample titrant})\,/\,(\text{ml of})$ 

std titrant).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample which will contain only one analyte per solution every three months labelling it with the date. The technical grade chromic acid weight percent is ideally 97-100 % for these raw materials.

# 4.19 RAW MATERIAL DETERMINATION OF TECHNICAL GRADE PHOSPHORIC ACID AND SULFURIC ACID BY ACID/BASE TITRATION USING A PH METER

This procedure describes the raw material determination of phosphoric acid and sulfuric acid by acid/base titration using a pH meter. References: (1, 5, 6).

Glassware includes: 10 ml pipets, 250 ml volumetric flasks, 25 ml pipets, 200 ml beakers, 50 ml buret, and 100 ml

volumetric flasks.

Reagent solutions include: 1.000 normal sodium hydroxide titrant, pH= 4.00 standard buffer one, pH= 10.00 standard buffer two, and a quality control reference sample of 50:50 percent mixture by volume of analytical reagent grade concentrated phosphoric acid and concentrated sulfuric acid each of known individual acid concentrations.

A pH meter, magnetic stirrer, and magnetic stirring bars are required. Precautions include only normal laboratory

practices.

Procedure for standards is not applicable. Procedure for samples is three replicates, pipet 25 ml of technical grade phosphoric acid and 25 ml of technical grade sulfuric acid into a 100 ml volumetric flask, add no water, mix and cool samples thoroughly, pipet 10 ml of a sample solution into a 250 ml volumetric flask rinsing pipet into flask with deionized water then filling to the mark with deionized water, cool and mix flask thoroughly, pipet 25 ml of the diluted sample solution in the flask into a 200 ml beaker, and fill the beaker to about the 100 ml mark with deionized water.

Procedure for sample titration is add a stirring bar to each beaker, calibrate the pH meter with buffer one (pH=4.00), put pH meter electrodes in the beaker and moderately stir, add the titrant to the buret as needed, dispense titrant from the buret until sample pH is 4.50+-0.10 pH units, record this initial titrant volume in ml dispensed as reading A, calibrate the pH meter with buffer two (pH=10.00), continue titrating until sample pH is 9.70+-0.10 pH units, and record the total titrant volume in ml dispensed as reading B.

The original sample calculations are grams/liter of phosphoric acid in the sample equals (2)(98)(sodium hydroxide titrant normality)(reading B - reading A) and grams/liter of sulfuric acid in the sample equals (2)(49)(sodium hydroxide titrant normality)((2 \* reading A) - reading B)).

Important notes are run a quality control reference sample with known concentration for each analyte just as if it were any other sample and remake this reference sample every three months labelling it with the date. The technical grade phosphoric acid and sulfuric acid concentrations are ideally 1280-1460 and 1590-1790 g/l for these raw materials.

## 4.20 COMPOSITE MATERIAL CHARACTERIZATION BY THERMAL ANALYSIS

This procedure describes the composite material characterization by thermal analysis. References: (1, 21).

Various glassware is required. Reagents used include: potassium bromide, tetrahydrofuran, acetone, epoxy resins, various curing agents, dimethylformamide, acetonitrile, ether, chloroform, alcohols, methyl ethyl ketone, and tetramethylurea.

Miscellaneous materials vary and precautions include only normal laboratory practices.

The procedure for samples follow. For differential scanning calorimetry, samples are weighed and sealed in aluminum pans for analysis. The samples are programmed to be heated either isothermally or dynamically up to 700 C. Sample types are primarily organic polymers such as epoxies or polyimides and weigh no more than 20 mgs. For thermogravimetric analysis, samples are placed in a thermobalance and then heated in a furnace up to 1000 C. Sample types are typically organic, ranging from polymers to oils and weighing no more than 50 mgs. For thermomechanical analysis, samples are placed on a platform and various types of probes are brought into contact with the surface. The samples are then heated up to 700 C and the interaction with the probe is measured. Sample types are organic polymers ranging up to 50 mgs in weight. For infrared spectrophotometry, samples types can be solids, liquids, or gases. The specimens can be analyzed in a cell as a solution with a solvent, ground and mixed with KBr then pressed in a pellet, smeared on a KBr plate, or pressed on both sides of a reflecting plate. Any sample that has a dipole can be analyzed and the sample size is a function of the technique used. Sample calculations vary.

### 4.21 OTHER INFORMAL CHARACTERIZATION METHODS

These procedures describe many other informal characterization methods that are used periodically. References and titles for these procedures are indicated. References: (1, 22-42). Glassware, reagents, materials, and precautions vary.

Procedure for samples include the following titles: The Analysis Of Metal Finishing Solutions By Ion Chromatography, Determination of Hexa-Aquo-Complexes of Chromium and Iron in Chromium Plating and Polishing Solutions by Ion Chromatography, Determination of Phosphoric, Sulfuric and Chromic Acids in the Presence of Matrix Effects for Chromium Plating and Associated Polishing Solutions by Ion Chromatography, Determination of Sulfuric and Oxalic Acids in the Presence of Matrix Effects for Aluminum Anodizing . Solutions by Ion Chromatography, Determination of Ethylene Glycol Degradation Products in Chromium Plating and Associated Polishing Solutions by Ion Chromatography, X-Ray Fluorescence On-Stream Analysis of Standard Reference Solution Concentrations of Chromium Plating and Polishing Solutions, Determination of Copper, Cadmium and Iron in Metal Cyanide Waste Solutions by Atomic Absorption Spectrometry, Determination of Sulfuric Acid in Chromium Plating Solutions Using Gravimetric Analysis, Chromium Plating And Electropolishing Solution Analyses By Online X-ray Fluorescence Spectroscopy, Determination of Trivalent Chromium in Chromium Plating Solutions Using a Redox Titration and Indicator, Determination of AISI 4340 and 8640 Steels Used In M9 Beretta Pistol Slides By Emission Spectroscopy and X-Ray Fluorescence Spectroscopy, Investigation Of Exothermic Grinding Sludge Produced From Watervliet Arsenal Gun Steels, Andersol Water Based Cutting Fluid And Cincinnati Milacron Aluminum Oxide Resin Bond Grinding Wheels, Determination Of PCB's In Watervliet Arsenal Machine Oils By Gas Chromatography, Laboratory Automatic Titration of Chromium Plating and Electropolishing Solutions, Chemical Analysis of 105, 120 and 155 mm "Gun Steels" By Emission Spectroscopy, Inductively Coupled Plasma Spectroscopy and Carbon/Sulfur Analyzer, Determination Of Sulfuric Acid In Chromium Plating Solutions By Turbidimetric Analysis, Automatic Coulometric Titration of Sulfuric Acid In Chromium Plating Solutions, Online Automatic Titration of Chromic Acid In Chromium Plating Solutions And Phosphoric And Sulfuric Acids In Electropolishing Solutions, Online Automatic Titration of Sulfuric And Chromic Acids In Chromium Plating Solutions, and Determination Of Wastewater Acids From Chromium Plating and Electropolishing Solutions. Sample calculations vary.

#### 5. STANDARD SAMPLING AND REPORTING METHODS

### 5.01 STANDARD SAMPLING METHOD

This procedure describes the sampling method that will be used for the Chemistry Lab. References: None

Procedures for sampling are all samples requiring chemical analysis by the Chemistry Lab will only come into the Chemistry Lab Sample Drop Point in Bldg 115. All samples left at the Chemistry Lab Sample Drop Point will be logged-in to the zsamples.dbf database file in capital letters as soon as possible. Refer to the next section for further information related to the Sample Database Input Form. All uncompleted logged-in samples will be stored in one common location. Completed samples are discarded. All samples requiring chemical analysis by contractors will only be picked up at the Chemistry Lab Sample Drop Point in Bldg 115 after they are entered into the zsamples database. Call the contractor for sample pickup.

#### 5.02 ZSAMPLES DATABASE INPUT METHOD

The zsamples database input method allows only capital letters for data entry. Instructions are given below for data entry into the fields of the database input form: labnum with yymmdd.## format, Datecom with yy/mm/dd format, project is monthly report project numbers, charge is monthly report charge number, hours are twice the lab time which is approximately the lab time plus the overhead time, redouble that time for either rush samples or unscheduled samples, custlnam is customer's last name, sampleid is customer's sample id, samtype is customer's sample type, analyst is analyst's three initials, datered with yy/mm/dd format, comment: analyst's comments, experimental data: enter data for all or part of the following nineteen fields: elem1 - elem12, Cro3, CrIII, Fe, H2SO4cp, H2SO4sd, H3PO4, and H2SO4ep.

#### 5.03 STANDARD SAMPLE DROP POINT FORM

The data on this form includes: date submitted, originator's last name, originator's organizational symbols, originator's charge number, sample id number, sample type/grade, and analyses required.

#### 5.04 STANDARD MONTHLY SCHEDULE

The current schedule includes information for sampling time, sampling source, sampling number, and sampling type.

1st, 2nd, 3rd & 4th Monday Sampling is from: SMCWV-OD (20 Misc. Solid Samples), SMCAR-CCB-S (8 Misc. Samples), SMCAR-CCB-R (8 Misc. Sample), and SMCAR-CCB-R (1 Misc. Composite Type).

Every Monday and Thursday Sampling (Liquid Samples) is

from SMCWV-OD (2, 8, 2P, 8P) for All Acids.

Additional 1st Monday Sampling (Liquid Samples) is from: SMCWV-OD (2, 8) for Cr3, SMCWV-OD (1, 27-30) for H2SO4, CrO3, Cr3, and SMCWV-OD (2, 6, 8, 9) for Ni, Dye or Cr.

Additional 2nd Monday Sampling (Liquid Samples) is from: SMCWV-OD (2, 8, 2P, 8P) for Fe, SMCWV-OD (1, 27-30) for H2SO4, CrO3, Fe, and SMCWV-OD (19, 20, 25, 26) for H2SO4.

Additional 3rd Monday Sampling (Liquid Samples) is from: SMCWV-OD (2, 8) for Cr3, SMCWV-OD (31-34) for H2SO4, CrO3, Cr3, and SMCWV-OD (19, 21, 30, 31) for CN & Cu or Cd.

Additional 4th Monday Sampling (Liquid Samples) is from: SMCWV-OD (2, 8, 2P, 8P) for Fe, and SMCWV-OD (31-34) for H2SO4, CrO3, Fe.

SMCWV-OD samples 2, 8, 2P & 8P have a 24 hour reporting time and are top priority. All other samples have a one month reporting time. Urgent SMCWV requests are coordinated through SMCWV-ODP-C and urgent SMCAR requests are coordinated through SMCAR-CCB-RT.

Work Execution Documents (WED's) are required before all sample characterizations. This schedule assumes a work month with no holidays, a full staff and overtime.

## 5.05 STANDARD PROCEDURE FOR REPORTING LIQUID SAMPLE RESULTS

This procedure describes the method that will be used to report all liquid sample results. References: None

Procedures for analyses are three replicates and use standard analytical methods to obtain these analytical data.

Procedures for liquid sample quality control data are reference samples will be obtained using the standard quality control methods for each liquid sample analyte. The first quality control method requires that a standard solution is ran as a reference sample for each analyte of that liquid sample. For the reference samples, put these values in the zsamples database with the sample ID as refsample. The second quality control method requires that two analysts each do two replicates each for sulfuric, phosphoric and chromic acids. For the reference samples, each person puts their own average values in the zsamples database with the sample ID as refsample.

The calculations and sample decoding are done in the z120 spreadsheet for the sulfuric acid in the chromium plating solutions two and eight. When a sample from either chromium plating solutions two or eight fails the t-test portion of the above spreadsheet then that sample is invalid, a new sample is requested and the analysis is redone. All other liquid sample data can be calculated using any available means.

Use of the zsamples database is also for all other liquid sample data which is entered directly into the database. All liquid sample quality control data for reference samples will be entered directly into the database for each liquid sample analyte.

Special required reports for 2, 8, 2P & 8P include the following. For liquid samples 2, 8, 2P and 8P: provide one copy of the database report form for each liquid sample to SMCWV-ODM-F, provide one copy of the database report form for each liquid sample to SMCWV-ODQ, and call SMCWV-ODQ at x5369 to leave word of any out of tolerance data for 2, 8, 2P and 8P.

Required reports for other liquid samples include one copy of the database report form for each liquid sample to the customer.

Distribution of reports are not done without approval from the lead technician.

Analyst's copies include backing up the zsamples database files weekly on diskette.

# 5.06 STANDARD PROCEDURE FOR REPORTING SOLID SAMPLE RESULTS

This procedure describes the method that will be used to report all solid sample results. References: None

Procedures for analyses are three replicates and use standard analytical methods to obtain these analytical data.

Procedures for solid sample quality control data are reference samples will be obtained using the standard quality control methods for each solid sample analyte. This quality control method requires that a standard is ran as a reference sample for each analyte of that sample. For the reference samples, put these values in the zsamples database with the sample id as refsample.

Calculations for solid sample results are done only by the attached computer/ microprocessor of the analyzing instrument.

Use of zsamples database for solid sample data will be entered in the database only from the printouts of the computer/ microprocessor of the analyzing instrument and all solid sample quality control data for standards will be entered directly into the database only from the computer/ microprocessor of the analyzing instrument for each solid sample matrix analyte.

Required reports include one copy of the database report form for each reported solid sample to the customer. Attach the Procurement inspection ticket were applicable.

Distribution of reports are not make without approval

from the lead technician.

Analyst's copies include backing up the zsamples database files weekly on diskette.

# 5.07 STANDARD PROCEDURE FOR SIGNIFICANT ACTIVITY REPORTS

This procedure describes the method that will be used for significant activity reports (SAR) to CCB-RT. References: ARDEC Format.

Procedure includes: division: SMCAR-CCB-R, date, subject: newspaper headline style, reportable item: newspaper article style, type of item: new, update, or special interest, poc, autovon number, and commercial number.

# 5.08 STANDARD PROCEDURE FOR REPORTING ORDERED MATERIALS

This procedure describes the method that will be used to report all ordered materials to CCB-RT. References: none

Procedure for method is all orders for chemicals must state that appropriate msds's are also required, the Chemistry Lab will not receive any chemical without its msds, hazardous material should be identified as such on any procurement orders, and the Chemistry Lab ordering officer is responsible for the zorder database on the Chemistry Lab's main computer. Specifically, this person will order for local purchases, SITs, ASSSC and 1095s with updates as needed, use the appropriate forms subscribed by CCB-RT for ordering items entered in the zorder database, and the ordering officer will backup the zorder database weekly.

# 5.09 STANDARD PROCEDURE FOR REPORTING CATALOGED STANDARD REFERENCE MATERIALS

This procedure describes the method that will be used to report all cataloged standard reference materials (srm's). References: none

The procedure for this method is the Chemistry Lab's srm cataloging officer is responsible for the zsrm database on the Chemistry Lab's main computer, this person will catalog all srm's used in the Chemistry Lab and update as needed and backup the zsrm database weekly.

# 5.10 STANDARD PROCEDURE FOR REPORTING CATALOGED HAZMAT'S AND MSDS'S

This procedure describes the method that will be used to report all cataloged hazmat's and msds's. References: none The procedure for the method is all chemical users please update the list on the appropriate cabinet as a container of a chemical is purchased, used up, etc, remember that each container is a separate database record even if two containers have the same contents, the Chemistry Lab hazmat and msds cataloging officer is responsible for the zhazmat database on the Chemistry Lab's main computer, this person will catalog all hazmat's and msds's used in the Chemistry Lab and update as needed and provide an updated copy of the hazmat inventory to the CCB-RT office 340a file as needed, msds binders are to be labelled using a label maker as follows: msds, bldg 115, vol #, do not remove, for each hazmat storage cabinet, the above officer will provide an updated monthly hazmat list of the cabinet contents and attach it to that cabinet as well as backup the zhazmat database weekly.

# 6. PROCUREMENT, HAND RECEIPT, AND SHIPPING INFORMATION

# 6.01 EXAMPLE OF A REQUIRED MEMORANDUM TO SMCWV-PPA FOR EQUIPMENT DEMONSTRATIONS

The body of this memorandum should contain information such as the following example. References: DF, SMCWV-PP, dated 23 March 19XX, Subject: Unauthorized Contracts with Vendors and Contractors. FONECON, S. Sopok, SMCAR-CCB-RT / E. Brownell, SMCWV-PPA, 02 Jan 19XX, Subject: Market Analyses. During the next three months, I will visit the applications laboratories of the Mettler Corporation (Hightstown, NJ), Brinkmann Corporation (Westbury, NY), Orion Corporation (Boston, MA), and Ionics Corporation (Watertown, MA) for an equipment demonstration. Each of these firms will perform a series of no-cost analyses of chemical samples from Benet Labs. I will explain to each firm that the demonstration is for market analysis purposes and in no way constitutes any current or future commitment on the part of the Government. I will contact Betty Brownell (Chief of SMCWV-PPA) or her alternate ((518) 266-5900) if a firm raises any issue of incurred cost.

#### 6.02 PROCUREMENT/WORK DIRECTIVE (1095) EXAMPLE

The description should contain information such as the following laboratory and online autotitrator examples. The specifications and price breakdown for the Mettler Model DL70 Lab Automatic Titration Analyzer follow. The total price of this system is about \$ 15,000. The system includes the Mettler DL70 Automation System, additional burette drive, additional 10 and 20 ml burettes, glass electrode, pH electrode, copper electrode, platinum electrode, reference electrode, ST 20 sample changer, Mettler two-year Service Plus Protection, FX850+ printer, rod stirrer, set of plastic beakers, and the IBM PS/2 model 50 data system with real-time titration monitoring. Mettler will provide revision level 2.0 of DL70 operating software at no additional cost as well as instrument manuals, programming, inspection, installation, startup assistance and training. Sources: Mettler Instruments Corporation, Hightstown, NJ (609) 448-3000. If Mettler Instruments Corporation did not have this instrument on the GSA list then three sources are required for procurement. GSA Source Statement: All Mettler Instruments Corporation Model DL70 System items have been accepted by the GSA under the current contract number GS-OOF-06802 except the IBM PS/2 model 50 computer. These GSA accepted items reflect the discounted price.

The specifications and price breakdown for a generic online automatic titrimeter follow. The total price of this model is about \$ 55,000 excluding transportation and state taxes. The system includes an online titration chemical analyzer including basic unit, tool kit, manuals, programming, industrial cabinet, 4-6 burettes/4-6 drives/4-6 sample loops/4-6 sample loop pumps (or any equivalent burette/sample loop combination equal to ten), colorimeter, reaction vessel, pH electrode, stirrer, copper electrode, platinum electrode, reference electrode, date time printer, ac line filter, and five reagent containers. Instrument spare parts kit, inspection, installation, startup assistance and training are also included. Sampling and analysis of suspended solids up to one-sixteenth of an inch is required. Transportation and expenses to be paid by customer. User to provide State Tax Exemption No. or state taxes will be added. Only two sources are available: Ionics Corporation, Watertown, MA, (617) 926-2500, Brinkmann Instruments Corporation, Westbury, NY, (516) 334-7500.

# 6.03 PROCUREMENT/WORK DIRECTIVE (1095) EXAMPLE (ANALYSIS CONTRACT)

The description should contain information such as the following example. The specifications and price breakdown for the chemical analysis contract include the following. The total price is about \$ 75,000. The contract includes: the provided analyses of 96 chromium plating solutions for Cro3, Cr(III), total Iron, and H2SO4 in g/l paid on a per sample basis, the provided analyses of 96 polishing solutions for H3PO4, H2SO4, and total Iron in g/l paid on a per sample basis, and the requirement of three significant figures for each analyte. The contract will run from 01 Mar 19XX thru 01 Apr 19XX. There will be four chromium plating solutions and four polishing solutions sampled at the Arsenal on both the first and third Mondays of each month for the given year. The vendor is to supply bottles, labels, and sample pickup. Sources include: Environmental One Corp., Schenectady, NY, (518) 346-6161, Adirondack Environmental, Rensselaer, NY, (518) 434-4546, and C. T. Male Assoc., Latham, NY, (518) 785-0976.

# 6.04 BLANKET PURCHASE AGREEMENT (LOCAL PURCHASE) EXAMPLE

Use DRDAR-LC Form 896 and the following items: to: ordering officer, Research Division, suggested source, date, items (no substitutes), company, address, units, cost, description, charge no, cost area, requester, and tel no.

# 6.05 WATERVLIET ARSENAL SIT EXAMPLE

Use SIT Form (SARWV 2034) and the following items: requires initials, description, justification, quantity, org.symbl, unit, est. unit cost, stock no, date required, job or no or date, o. o. no. is 0705, acct. no. is 388, written by, org, and telp.

## 6.06 WATERVLIET ARSENAL ASSSC EXAMPLE

Use DA Form 3161, DA PAM 710-2-1, and blue Automated Self Service Supply Center (ASSSC) Administrative Supply Catalog. Information includes: questions (x5444 or x5451), location and pickup hours (bldg 23, 2nd floor, Tuesday, Thursday, 0745-1130 and 1200-1500), bring CCB-RT's credit card, and bring Form 3161 with the following for each item: item no., stock no., item description, unit issue, no., cost, date, and by.

#### 6.07 HAND RECEIPT EXAMPLE

This example consists of two parts. Use WVAR Form 710-4 or SMCWV Form 2104 for part 1 issue or turn-in including the items of hand receipt number, date, qty, requested, unit price, total cost, nomenclature, justification, fund citation, cost charge, typed name and title. Use DA Form 710-2-1 or DA Form 2765-1 for part 2 issue or turn-in including the items of send to SMCWV-LDE, unit, quantity, cost detail account number, unit price, total price, item description, and publication data.

### 6.08 REQUEST FOR SHIPMENT EXAMPLE

An example for hardware and an example for chemicals is enclosed. For the hardware example, use SMCWV Form 2023 or WVAR Form 735-7 that includes the items of initials s.o, to SMCWV-ODS-LA, bldg 25 Supply Div, request no, from, request date, signature, required ship. date, authority, priority, ship to, mark for, charge transportation, pck. & handling, item nr, nomenclature, unit, qty, unit price, total cost, and special instructions such as packing, preserving, crating, or other, milstrip codes.

For chromium plating process solutions include information such as the following example: ten foam encased glass bottles each containing 475-500 ml of chromic acid solution 25% by weight-approximate (UN1755), and ten foam encased glass bottles each containing 475-500 ml of sulfuric acid 50% by approximate weight and phosphoric acid 50% by approximate weight and phosphoric acid 50% by approximate weight solution (UN1760). Items are shipped in twenty individual foam encased bottles (individual mailers included) and each item weights less than 15 pounds.

#### 7. PROJECT PLANNING AND REPORTING INFORMATION

#### 7.01 CHEMISTRY LAB PROJECT PLANS EXAMPLE

Statement of work/objective: Advanced Technology Branch's Chemical Lab (SMCAR-CCB-RT) will provide about 3.0 man-years during FYXX for characterizations of chemical processes and materials and consultation for Operations Directorate (SMCWV-OD).

Program: The strategy followed and major actions taken are given in the above FYXX standard monthly schedule and the degree of achievement of these objectives/milestones will be formally reported monthly.

Schedule: Individual and group activities/accomplishments will be started and completed according to the Chemical Lab's FYXX standard monthly schedule and its individual standard work schedules.

Budget: Planned expenditures required to achieve direct labor objectives include 3.20 man-years for 3.20 chemical technicians including overtime authorized at 0.20 man-years. Planned expenditures required to achieve contract/material objectives include \$70,000.

Forecast: Projections of what will happen by certain times are again given in the Chemical Lab's FYXX standard monthly schedule.

Organization: The design includes 3.20 chemical technicians. Their corresponding duties and responsibilities required to achieve or exceed objectives are given in their Federal job descriptions.

Policy: A general guide for decision making and individual actions are set and approved by the Chief of the Advanced Technology Branch (SMCAR-CCB-RT) and Federal regulation.

Procedure: A detailed method for carrying out a policy is set and approved by the Chief of the Advanced Technology Branch (SMCAR-CCB-RT) and Federal regulation.

Standards: A level of individual and group performance defined as adequate or acceptable is given in their Federal job standards set and approved by the Chief of the Advanced Technology Branch (SMCAR-CCB-RT) and Federal regulation.

Labor cost/hour: This includes hourly rate, plus overhead at \$21 each, plus fringes at 40% for each hour worked. Only hourly rate and fringes at 40% are applied to ISA work, One year has 260 days or 2080 hours per person.

## 7.02 STANDARD INDIVIDUAL WORK PLANS

Enclosed are work plans for one project leader/lead lab technician and two working lab technicians. The work plans for the project leader/lead lab technician includes project management and monthly project reports.

The work plan for the first working lab technician includes: a monthly notebook report, quality work, cross-training and the following schedule: for Monday, cannon chromium plating process solutions analysis and reporting (highest priority), for Tuesday, small components plating process solutions analysis and reporting, for Wednesday, test all eyewash and shower stations, prepare solutions, clean lab area, for Thursday, cannon chromium plating process solutions analysis and reporting (highest priority), for Friday, small components plating process solutions analysis and reporting.

The work plans for the second working lab technician includes: a monthly notebook report, quality work, cross-training and the following schedule: for Monday, low alloy steel solid sample analysis and reporting, for Tuesday, non-low alloy steel solid sample analysis and reporting, for Wednesday, test all eyewash and shower stations, prepare solutions, clean lab area, for Thursday, low alloy steel solid sample analysis and reporting, and for Friday, non-low alloy steel solid sample analysis and reporting.

#### 7.03 PROJECT PROPOSAL EXAMPLE

Enclosed is a typical project proposal example which also includes the proposer's name and proposal date.

Project Title: Characterization Of Critical Low Concentration Constituents In Chromium Plating Processes.

Estimated Funding: Salaries 45K, OGA OK, Contracts 4K, Total 49K.

Goal: Characterize critical low concentration constituents (sulfuric acid, trivalent chromium, and iron) with an online automatic titration system for Benet Laboratories and Watervliet Arsenal plating facilities.

Background: Previous work on the online characterization of chromium plating processes by this investigator has included R&D efforts on ion chromatography, x-ray fluorescence, and automatic titration systems. The online automatic titration system is accepted as the most practical at this time. Characterization methods have been fully developed using online automatic titration for all major constituents in chromium plating (chromate) and electropolishing (phosphate, sulfate) solutions. Characterization method have been somewhat developed and require further effort for all critical low concentration constituents in chromium plating (sulfuric acid, trivalent chromium, iron) and electropolishing (iron) solutions.

Scope of Work: Develop method for sulfuric acid in chromium plating solutions, develop method for trivalent chromium in chromium plating solutions, develop method for iron in chromium plating solutions, develop method for iron in electropolishing solutions, evaluate data, and write technical report.

Application or Benefit: The full development of these characterization methods for all constituents by online automatic titration will contribute to more efficient cannon related production and environmental thrust programs. Specific applications and benefits include: reduced cannon production downtime, reduced QC and PAD data handling manpower, reduced EP cell operation, reduced outside contract chemical analysis, increased waste stream monitoring, increased process and quality control, possible future automated chemical additions, and increased hazardous waste minimization systems.

#### 7.04 GENERAL WORK PROFILE FOR THE CHEMISTRY LAB

Enclosed is a general work profile for the Chemistry Lab which outlines the types of work being applied to U.S. Army's needs. There has been a progression from "wet chemistry" to advanced instrumentation in the last ten years requiring a hundred thousand dollar annual Chemistry Lab modernization effort and nearly a million dollar annual total budget. As a result, characterization times have progressed from days to hours with increased sensitivity and equal accuracy.

Online and laboratory characterization efforts include: solid metal samples for/from ordnance, metal finishing and associated wastewater sample solutions for ordnance, composite samples for/from ordnance, thermodynamic and kinetic studies for ordnance.

## 7.05 CHEMISTRY LAB INVESTIGATIONS OF US ARMY HARDWARE

Chemistry Lab investigations of Army hardware include cannon/gun tubes, mortar tubes, mounts, muzzle brakes, rounds, charges, breeches, pistols, propellants, etc. for the following ordnance: 20 mm, 40 mm, 60 mm (M2, M225), 60 mm M224, 81 mm M29A1, 84 mm (M2, M3), 105 mm (XM872), 105 mm (M68, M68A1), 105 mm Howitzer (M2A2, M137A1, M20A1), 105 mm EX35, 120 mm (M256, M2256), 120 mm M285, 120 mm M820, 120 mm (NDI), 120 mm XM25, 155 mm HIP, 155 mm Howitzer (M185, M199, M284, M1A2), 155 mm M483A1, 155 mm Howitzer (M185, M284, M199), 155 mm X52 Caliber, 165 mm M135, 4.2 inch M30, 8 inch Howitzer (M201A1, M201), 8 inch M650, 16 inch (Navy), AEI Program, AFAS Program, AMAGS Program Anodes (105mm, 120mm, 155mm), ATACS Program Blake thermochemistry codes, Bore Evacuators (105mm, 120mm), Coatings (Al, Ni, Refractory Sputtered), Desert Shield/Storm Hardware Failures, Electro-Composites Program, Electro-Thermal Cannon Program, EM Launcher (Rail Gun) Program, Fused Salt Program, Gun Steels (Picatinny, Watervliet, Rock Island), HICAP Program, High Density Penetrators, HIP Program, Interior Ballistics (Thermal Management Program), LC Chrome/Vessel Plating Program, Liquid Propellants, LWIFS Program, M109 Program, M140 Pistol Program, M9 Beretta Pistol (PM-M9), MICLIC Switches, Miser (AT4) Program, Online Chemical Analyzers (Benet Labs, Watervliet), Organic Composites (Tow Bar, Cannon), Plating Solutions (Picatinny, Watervliet, Rock Island), Propellant Chemistry, RARDE Program, Refractory Liners, RLPG Program, Ta Liner (105mm, 155mm), TOPCAT Program, Unicharge Propellants, WVA FMA Hazardous Static Problem, WVA Grinding Sludge Fires, XM218 .50 Cal Gun, XM291, XM91, XM900, and XM900E1.

# 7.06 OUTLINE OF BENET LABS CHEMICAL/PHYSICAL TESTS FOR THE 120 MM CANNON

Enclosed is an outline of Benet Labs' chemical and physical tests for the 120 mm cannon and this information was recently requested by the Watervliet Arsenal for the Egyptian Technology Transfer Program. The outline is divided into three material classes consisting of solutions, metals and coatings.

The tests include: ion chromatography or ic, titration or t, visible spectrophotometry or vs, atomic absorption spectroscopy or aa, emission spectroscopy or es, inductively coupled plasma spectroscopy or is, carbon and sulfur analyzer or cs, and x-ray fluorescence spectroscopy for unknowns or xf.

Tests for chromium plating solutions include: sulfuric acid: ic, chromic acid: t, trivalent chromium: vs, iron: aa. Tests for electropolishing solutions include: phosphoric acid: t, sulfuric acid: t, and iron: aa.

Tests for anodize/hardcoating solutions include sulfuric acid: t, tests for nickel plating solutions include nickel: aa, tests for black dying solutions include black dye: vs, tests for olive dying solutions include olive dye: vs, tests for sealing solutions include sodium dichromate: vs, tests for copper plating solutions include copper: aa and cyanide: t, tests for cadmium plating solutions include: cadmium: aa and cyanide: t, tests for metal alloys include: iron: aa,es,is,cs,xf, copper: aa,es,is,xf, nickel: aa,es,is,xf, and aluminum: aa,es,is,xf, and tests for coatings include: chromium: aa,is,xf, nickel: aa,is,xf, copper: aa,is,xf, cadmium: aa,is,xf, and tantalum: aa,is,xf.

### 7.07 STANDARD MONTHLY REPORT EXAMPLE

The heading includes the following information: Project Leader, Project Number, Sales Order Number, Project Area, Project, Reporting Period, Customer, Category, Value Total, Planned and Authorized Value Current FY. The sub-heading includes: Description, Meeting Performance Requirements, On Schedule, Adequate Funding, Contracts.

The body includes Project Highlights such as: Progress, Problem Areas, Recommended Action/Action Taken, Prepared by, Date. Additional enclosures include: Gantt Chart, List of Current Contracts, List of Samples Completed, and List of Samples Uncompleted.

### 7.08 CHEMISTRY LAB CAPABILITIES/EQUIPMENT

Chemistry Lab capabilities include support of most research, development, engineering and production applications. Lab personnel are experienced in a broad range of inorganic, organic, physical, and analytical chemistry analyses using the modern equipment listed below.

Equipment descriptions are included for a vast array of spectrometers, chromatographs, thermal analyzers and other major analytical chemistry instrumentation. Other standard analytical chemistry equipment include pH meters, water purification systems, furnaces, and analytical balances.

The HP 8452 Diode Array UV/Visible Spectrophotometer and its HP 89500 computer/software are applicable to most inorganic and organic solutions. The technique is absorption spectrometric analysis of molecular and ionic chemical species in solution. Its features include single component, multiple component and kinetic analyses from 190 - 900 nm. This diode array type instrument samples spectrum up to twenty times a second.

The Angstrom V-70 Emission Spectrometer and its DEC PDP-11 computer/ software are applicable to iron, copper and aluminum metal alloys. The technique is emission spectrometric analysis of elemental chemical species in metals. Its features include chemical analysis of up to fifty elements for most types of metal samples. Chips or turnings from a machining operation can be melted in a special vacuum furnace and analyzed by this instrument.

The Dionex 2020i Ion Chromatograph and its Spectra-Physics computer/ software are applicable to metal finishing and wastewater solutions. The technique is chromatographic analysis of molecular and ionic chemical species in solution. Its features include the autosampling and chromatographic separation of chemical species followed by conductivity, uv/visible, and/or electrochemical detection of these chemical species. Due to a noncorrosive plastic flow stream, emphasis is on corrosive inorganic sample solutions.

The PE 1420 Double Beam Infrared Spectrophotometer and its Perkin-Elmer computer/software are applicable to organics such as polymers, oils and solvents. The technique is absorption spectrometric analysis of molecular and ionic chemical species in solution. Its features include an operating range of 4000 - 600 wavenumbers with scanning rates of 3 and 15 minutes.

The HP 1095 Liquid Chromatograph and its Hewlett-Packard computer/ software are applicable to polymeric and organic solutions. The technique is chromatographic analysis of molecular and ionic chemical species in solution. Its features include chromatographic separation of chemical species followed by diode array uv/visible and/or refractive index detection of these chemical species. Due to a stainless steel

flow stream, emphasis is on noncorrosive organic sample solutions.

The Leco CS125 Carbon/Sulfur Analyzer and its Leco computer/software are applicable to carbon and sulfur analyses in metals. The technique is induction heating/absorption spectrometric determination of elemental carbon and sulfur in metals. Its features include simultaneous determinations of carbon and sulfur in metals using oxidative induction heating and dual infrared detection. In additional, an electronic balance automatically compensates for sample weight and detection limits are to 0.001 weight percent.

The PE 5000/6500 AA/ICP Spectrometer and its PE 7300 computer/software are applicable to most metallic ionic solutions. The technique is absorption or emission spectrometric analysis of elemental chemical species in metallic ionic solutions. Its features include autosampling and both the emission and absorption modes from 170 - 800 nm for elemental analysis of transition metals. These transition metals must be put in solutions but otherwise the technique is straightforward.

The Dupont 903 Moisture Evolution Analyzer and its microprocessor are applicable to the trace determination of water in most solid samples. The technique is induction heating/electrochemical determination of moisture (H2O) in solids. Its features include sensitivity to 0.1 mg with a precision of about 2 percent.

The PE DSC7 Differential Scanning Calorimeter and its PE 7500 computer/ software are applicable to polymeric and composite materials. The technique is thermal analysis and thermodynamic analysis of solids and liquids. Its features include heats of reaction, phase transitions, thermal stabilities and heat capacities determined for solid and liquid samples. The temperature ranges used are from ambient to 750 C and the scanning rate is from 0.1 to 200 C per minute. Cure and glass transition temperatures are examples of thermodynamic data acquired from polymeric samples.

The PE TGA7 Thermogravimetric Analyzer and its PE 7500 computer/software are applicable to polymeric and composite materials. The technique is thermal analysis and thermogravimetric analysis of solid samples. Its features include thermal stabilities, rates of reaction, reaction processes and sample compositions determined for solid samples. The temperature ranges used are from ambient to 1000 C and the scanning rate is from 0.1 to 200 C per minute. Thermal decomposition data and fiber content are examples of thermogravimetric data acquired from composite material samples.

The PE Sigma 300 Gas Chromatograph and its Perkin-Elmer computer/software are applicable to organic species in solution. The technique is chromatographic analysis of molecular and ionic chemical species in solution. Its features include chromatographic separation of chemical species

followed by thermal conductivity or electron capture detection of these chemical species. The temperature ranges used are from ambient to 450 C and the scanning rate is from 0.1 to 10 C per minute.

The PE Thermomechanical Analyzer and its PE 7500 computer/software are applicable to polymeric and composite materials. The technique is thermal analysis and thermomechanical analysis of solid samples. Its features include thermomechanical stabilities determined for solid samples. The temperature ranges used are from ambient to 700 C and the interaction with the probe is measured. Thermomechanical data are acquired from polymeric and composite samples.

The PE FTIR Infrared Spectrometer and its PE 7500 computer/software are applicable to solvent, organic, polymeric and composite materials. The technique is spectroscopic analysis of gases, liquid and solid samples. Its feature include 4000 - 600 wavenumbers operating range and down to a fraction of a second scanning rate.

The Mettler DL70 Laboratory Automatic Titrator and its IBM PS2 computer/ software are applicable to metal finishing and wastewater solutions. The technique is automatic titration and analysis of molecular and ionic species in solution. Its features include pH, redox, specific ion electrode, electrochemical, spectroscopic, and autosampling methods of these chemical species.

The Ionics DigiChem 3000 Online Automatic Titrator and its microprocessor/ software are applicable to metal finishing and wastewater solutions. The technique is online automatic titration and analysis of molecular and ionic species in solution. Its features include pH, redox, specific ion electrode, electrochemical, spectroscopic, and online sampling methods of these chemical species.

The Brinkmann Applikon 2020 Online Automatic Titrator and its microprocessor/ software are applicable to metal finishing and wastewater solutions. The technique is online automatic titration and analysis of molecular and ionic species in solution. Its features include pH, redox, specific ion electrode, electrochemical, spectroscopic, and online sampling methods of these chemical species.

#### 8. CONCLUSION

This manual should provide a useful starting point for responsible and efficient Chemistry Laboratory operations in the future. The key to success is a quality product.

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